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MARCH 29, 1943
VOLUME 32, No. 7

AMERICAN DYESTUFF REPORTER

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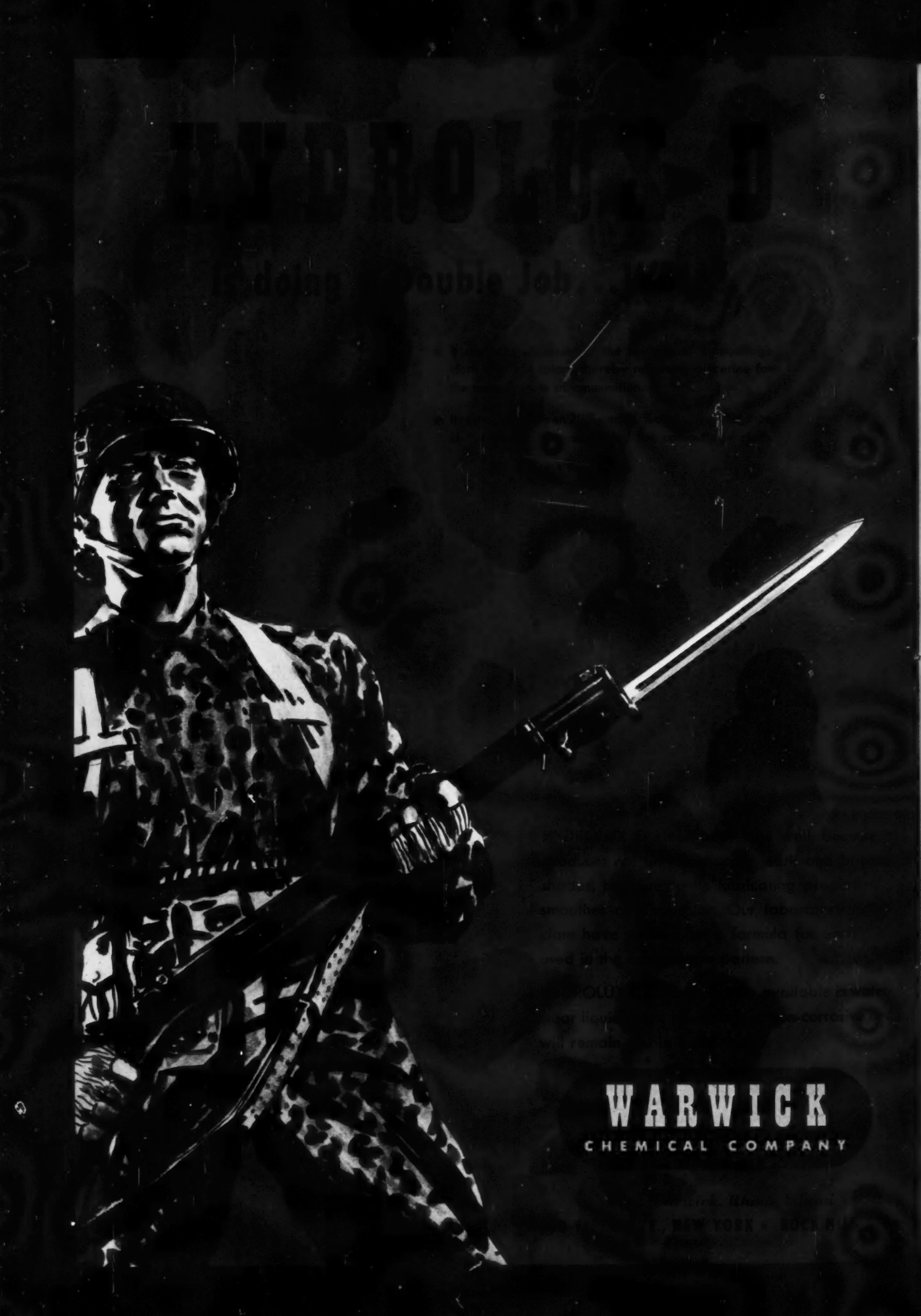
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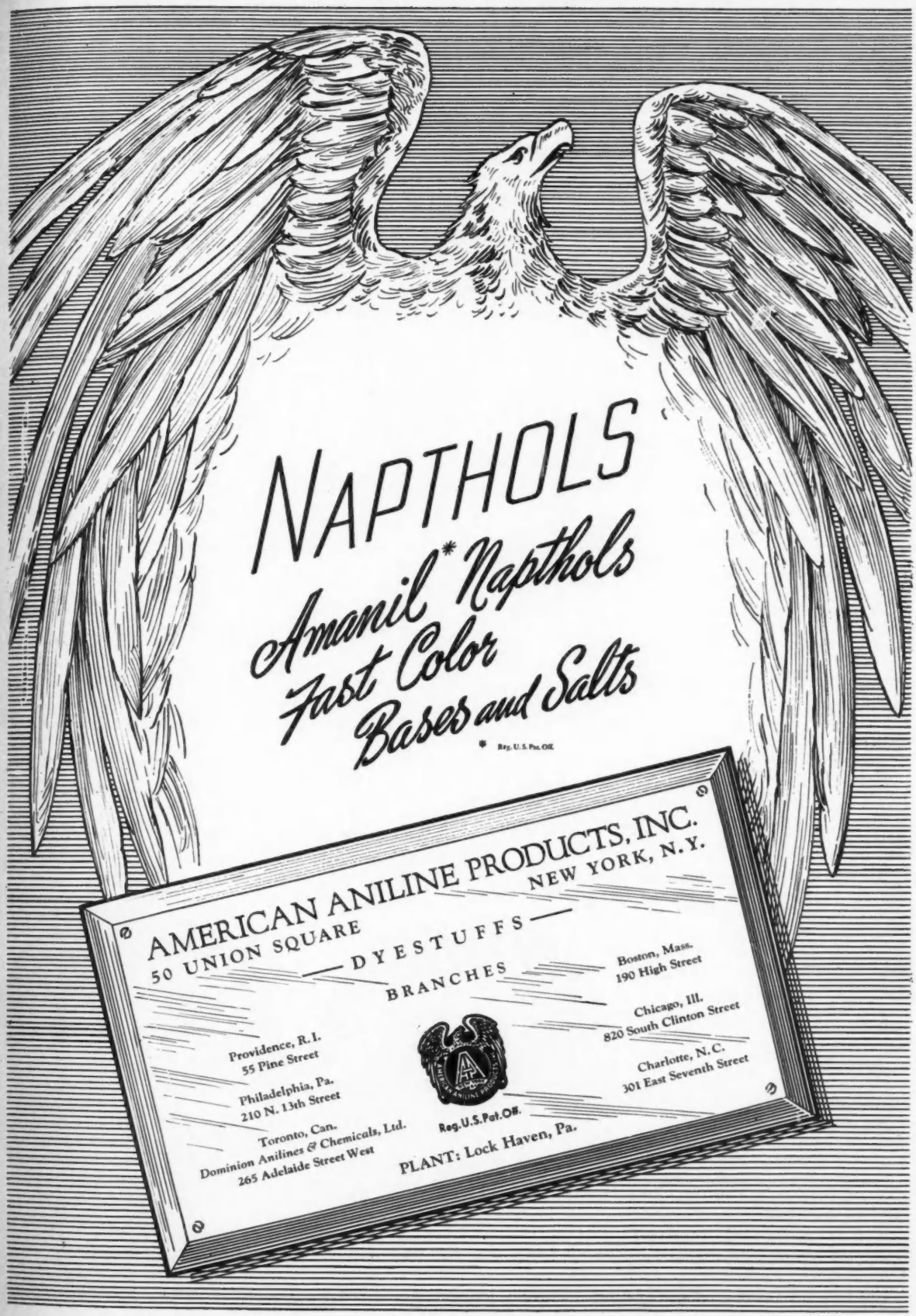

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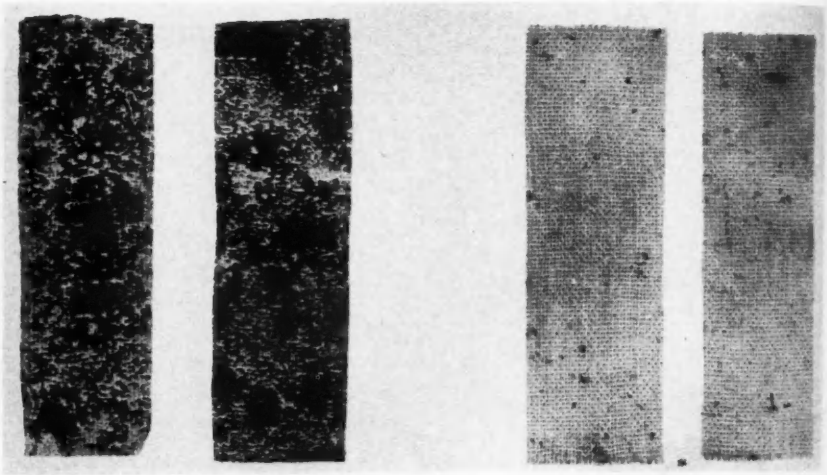

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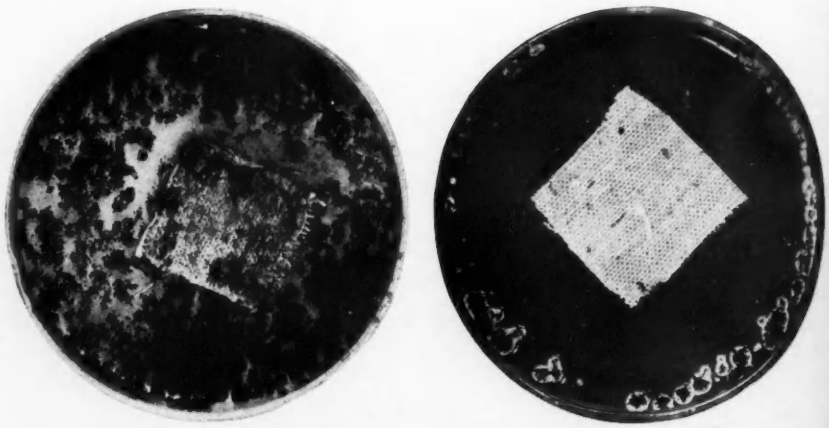

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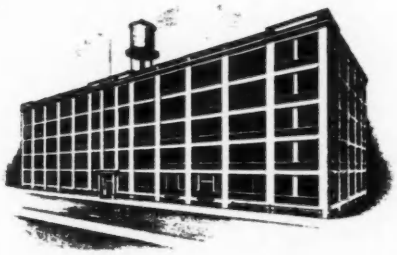

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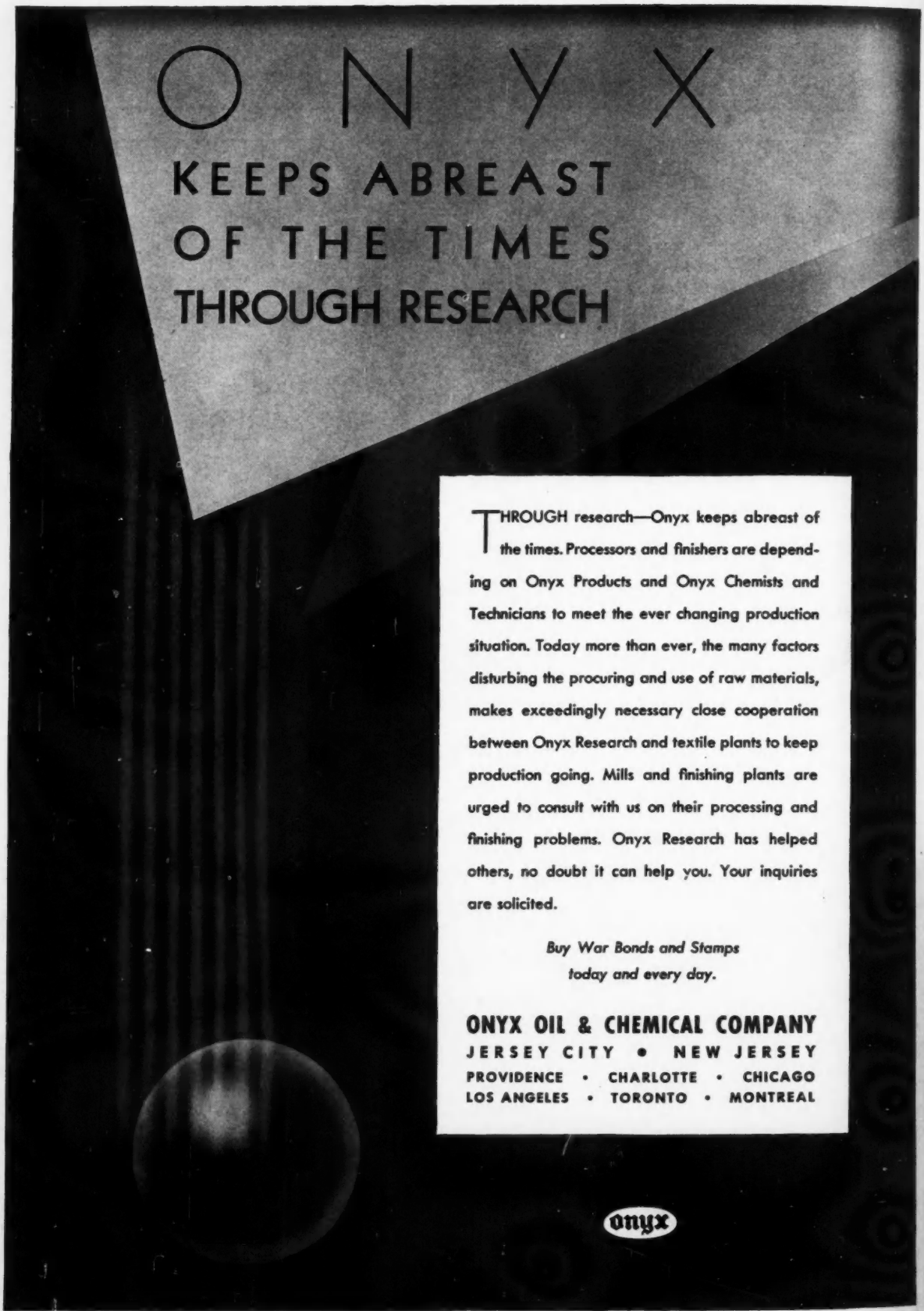

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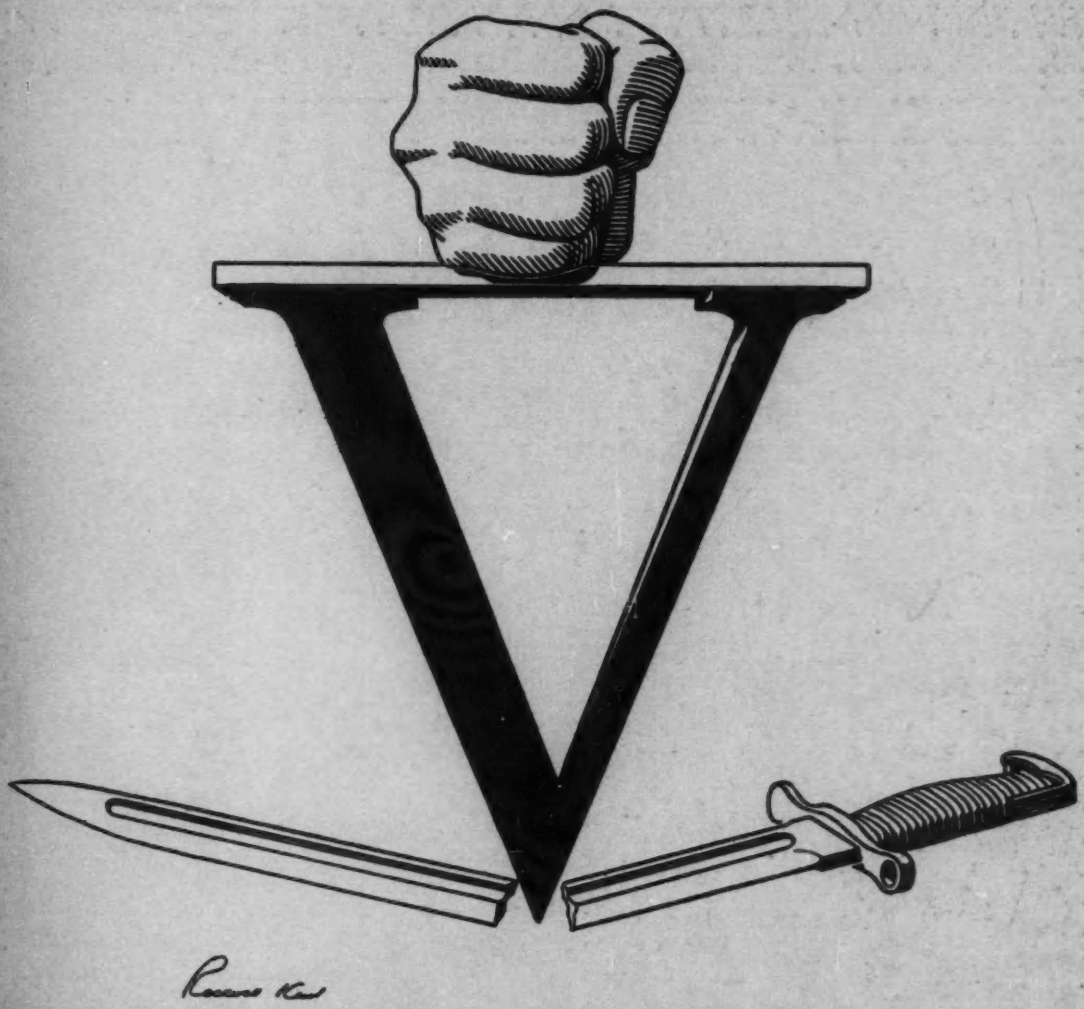

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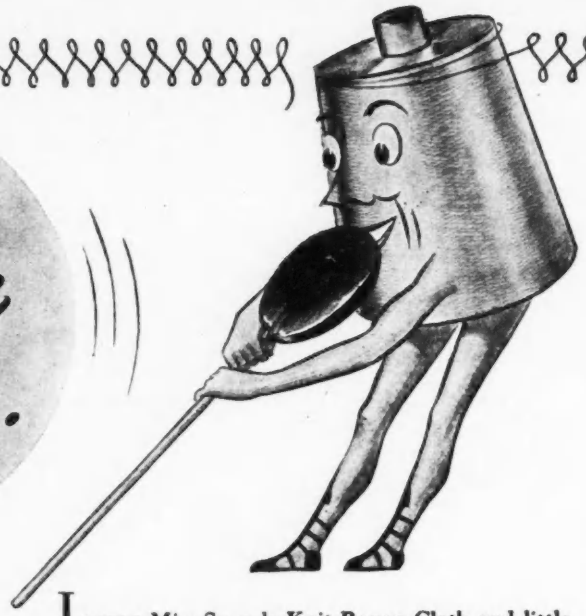

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NOT ONE, NOT TWO, BUT

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MARCH 29, 1943

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Contents

Contents of previous issues can be found by consulting the Industrial Arts Index at your library

• Special Articles

Nature of the Reaction of Wool with Alkali..... 145
Louis R. Mizell and Milton Harris

Heating Exhaust Steam..... 148
W. F. Schaphorst

Recent Advances in Fur Dyeing..... 163
Dr. H. Weisskopf

Microscopical Identification of Lanital, Aralac and Soybean Fibers..... 165
G. L. Royer

• Departments

Trade Notes—New Products..... 161

• Proceedings of the American Association of Textile Chemists and Colorists

Chemical Finishes for Rayon Fabrics..... P150
Dr. Hugh H. Mosher

Dyeing of Rayon and Some New Developments..... P153
James Hopwood

Meeting, Piedmont Section..... P156

Membership Applications..... P157

Calendar of Coming Events..... P160

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VOLUME 32 MARCH 29, 1943 NUMBER 7

Nature of the Reaction of WOOL WITH ALKALI

LOUIS R. MIZELL and MILTON HARRIS

Abstract

One of the most characteristic chemical properties of wool is the ease with which it is degraded in alkaline solutions. The effect of alkalies on wool has accordingly received considerable attention over a period of years by Research Associates of the Textile Foundation at the National Bureau of Standards. The present paper reports the results of further work in this direction.

Confirming earlier findings, it was found that such degradation is closely associated with the destruction by alkali of the disulfide groups of the cystine of the wool. Since these groups act as cross-links in the molecular structure of wool, thereby contributing considerably to the wet strength and to the long-range elastic properties of the fiber, the necessity for keeping them intact is at once obvious.

The reaction of the disulfide groups with alkali results in the rupture of the cross-links followed by the loss of one sulfur atom from each molecule of cystine. It appears that at least a part of the residual sulfur groups in the ruptured cross-links are capable of forming a new type of cross-link, thus partially stabilizing the system.

I. INTRODUCTION

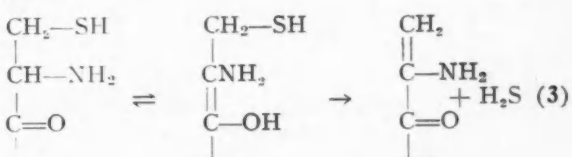
ONE of the most characteristic chemical properties of wool is the ease with which it is degraded in alkaline solutions. In earlier investigations[1, 2, 3] it was shown that such degradation is closely associated with the lability towards alkalies of the disulfide group in the cystine of the wool. A study of the course of the degradation revealed that during the early stages, a rapid splitting off of a portion of the sulfur occurred, the amount of sulfur being lost closely approaching 50 per cent of the original total[2]. This behavior led to the conclusion that the first step in the alkali degradation of wool is a splitting of the disulfide group to yield one labile and one comparatively stable sulfur group. Such a conclusion appeared to agree well with a mechanism that had been advanced by Schöberl and Eck[4] to explain the lability of sulfur in various disulfides. According to these investigators, the cleavage of disulfides by alkali consists in a hydrolytic rupture of the disulfide linkage with the formation of a sulfhydryl compound and a sulfenic acid according to the following scheme:

$$R{-}CH_2{-}S{-}S{-}CH_2{-}R \rightarrow RCH_2SH + HOSCH_2R \quad (1)$$

The sulfenic acid, being extremely reactive and unstable in alkaline solution, immediately loses H_2S to form an aldehyde as follows:

$$RCH_2SOH \rightarrow RCHO + H_2S \quad (2)$$

Another possible mechanism which was given consideration at that time was that proposed by Nicolet[5] who postulated that the removal of sulfur from cysteine by alkalies takes place as an elimination of the hydrogen sulfide from an intermediate enolized form according to the following equation:



(3)

He further suggested that from cystine, H_2S_2 would split out. Since this mechanism obviously failed to explain the loss of nearly half of the sulfur, little further attention was given it.

While Schöberl's hypothesis was consistent with the loss of half of the sulfur, it failed to explain a number of other observed facts. For example, his mechanism calls for the production of sulfhydryl and aldehyde groups, whereas tests for each of these groups on hydrolysates of the alkali-treated wool were definitely negative. As pointed out earlier[2], there existed the possibility that

recombination between the aldehydic and sulfhydryl groups may have occurred under the conditions of the hydrolysis. However, it has been subsequently shown[6] that few if any free sulfhydryl groups are present in the alkali-treated wool even prior to hydrolysis. In addition, this hypothesis, when applied to wool, would result in the rupture of the disulfide cross-links, a process which would be expected to yield a material of very low strength and extremely high alkali-solubility[7]. Yet samples of wool, treated with alkali by methods described elsewhere exhibit relatively high strengths and low alkali-solubilities (see Table I).

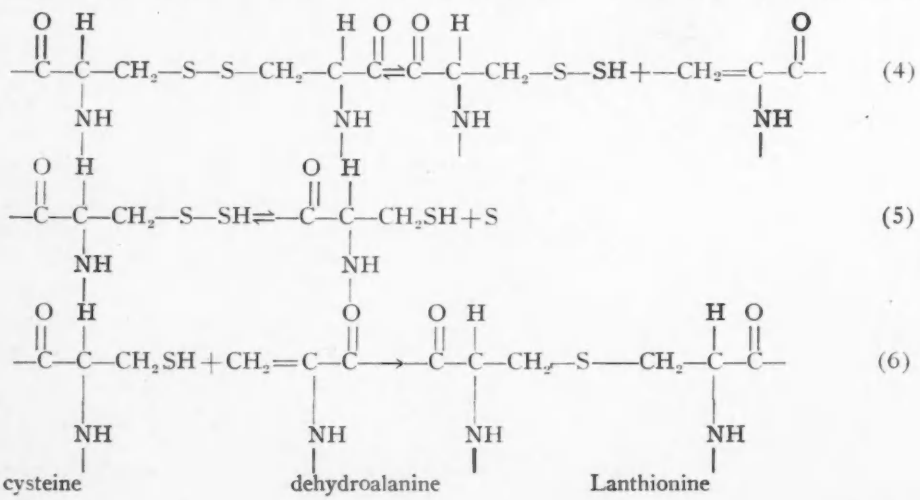
TABLE I
Effect on Wool of Treatment with Alkali by the Flow Method

Sample	*Cystine* *Found* %	*Calc.** %	*Total sulfur* %	*Alkali-solubility* %	*Serine* %
0.1 *N* NaOH flow for 600 hours at 0° C.					
Untreated	11.3	....	3.50	10.5	9.5
Treated	4.4	3.1	2.40	5.0	9.4
Treated + $C_6H_5CH_2Cl$.	4.2	....	....	....	....
0.1 *N* NaOH flow for 8 hours at 50° C.					
Untreated	12.5	....	3.50		
Treated	2.7	1.1	1.98		
Treated + $C_6H_5CH_2Cl$.	2.5	....	....		

* Calculated on the basis that each atom of sulfur lost by the wool represents the destruction of one molecule of cystine.

In fact, the properties of the alkali-treated wools are such as to strongly suggest that if the disulfide cross-links are destroyed, they are in a measure replaced by new linkages.

Considerable support for the view that new linkages can replace ruptured ones was found in the recent work of Horn, Jones, and Ringel[8] who succeeded in isolating from alkali-treated wool, a sulfur containing amino acid, having the formula $(HOOC—CH(NH_2)—CH_2)_2S$ which they named lanthionine. Since practically all of the sulfur in undegraded wool can be accounted for as cystine sulfur, it seems certain that this new amino acid is a product of the reaction of the cystine in wool with alkali. More recently Nicolet and Shinn[9] have offered an explanation for the formation of lanthionine postulating as a primary reaction, a split between carbon and sulfur as follows:



(4)

(5)

(6)

Such a mechanism has many advantages over the Schöberl hypothesis since it not only explains the loss of half of the sulfur, but in addition, it meets the requirement for linkage rebuilding and would thus account for the relatively high strength and low alkali-solubility of alkali-treated wools. Finally, it would explain the absence of significant amounts of sulfhydryl and aldehyde groups in the wool hydrolysate.

On the basis of the new point of view resulting from the work of Horn, Jones and Ringel and by Nicolet and Shinn, it appeared desirable to reinvestigate the course of the reaction of wool with alkalies.

II. EXPERIMENTAL PROCEDURE

1. Preparation of Alkali-Treated Wools

The low reported yields of lanthionine, obtained from alkali-treated wools (about 5 per cent[8]), offer some objection to the immediate acceptance of the hypothesis of Nicolet and Shinn. While it is recognized that values obtained by isolation methods are always low, nevertheless, it should be possible to demonstrate the formation of large amounts of this amino acid in the alkali-treated material of the reaction proceeds as suggested. On the basis of earlier work[2], it would appear that sodium sulfide, one of the products of the reaction of wool with alkali, could interfere with the formation of lanthionine in either of two ways. It might compete with cysteine in the reaction with dehydroalanine (eq. 3) or, it could split unreacted disulfide linkages and thus lower the amount of dehydroalanine that could be produced.

In order to overcome this difficulty, resort was had to a flow method, in which a fresh solution of alkali, either at 0° C. or 50° C, was allowed to flow continuously over the wool. Thus the reaction products were constantly washed from the fibers. In addition, by working at these lower temperatures instead of at the boil, secondary reactions would be kept at a minimum. At the lower temperature, the treatment was performed in a 12-liter flask which was immersed in a water bath at 0° C. The flask was fitted with an inlet and an outlet tube. 350 g of wool yarn were placed in the flask and 0.1 *N* NaOH, precooled to 0° C, was continuously flowed over the wool for 600 hours at a rate of about 2 liters per hour. In the experiment at 50° C, 133 g of wool were placed loosely in a cheesecloth bag, attached to a plunger, and agitated in a monel metal cylinder which contained about 10 liters of 0.1 *N* NaOH solution at 50° C ± 2. Fresh solution at the same temperature was continually siphoned into the bottom of the

cylinder at a rate of 12 liters per hour for 8 hours. The liquid in the cylinder was maintained at a constant level by removing the old solution as rapidly as the fresh was introduced.

At the end of each treatment, the samples were removed from the alkali and washed with distilled water. A sodium nitroprusside test, used for the qualitative detection of sulfhydryl groups, was negative on both samples. Immediately after the washing treatment, small portions of the wools were treated with benzyl chloride[7] in order to combine any cysteine residues that might be present.

2. Analytical Procedures

Cystine analyses were made by the Sullivan method[10], sulfur analyses by the oxygen-bomb method[11], alkali-solubilities by the method of Harris and Smith[12], and serine analyses by the method of Nicolet and Shinn[13].

3. Isolation of Lanthionine

Lanthionine was isolated from the alkali-treated wool by a slight modification of the procedure described by Horn, Jones and Ringel[8]. The modification was used to remove residual cystine from the residue containing the water-insoluble amino acids and the method was carried out as follows. The residue was taken up in 150 ml. of water and concentrated ammonium hydroxide was added dropwise until the residue was completely dissolved. 1.5 g. of sodium cyanide was then added to the solution and the mixture was allowed to stand 1 hr. after which it was made acid to litmus by the addition of acetic acid. The solution was kept in a refrigerator for 48 hrs. and the crystals which formed were filtered off. The filtrate was concentrated in vacuo and allowed to stand in the refrigerator for 3 days and the crystals again collected. A third batch of crystals was obtained by again concentrating the filtrate. The crystalline precipitates were combined and further purified by suspension in 50 ml. of water and adding concentrated NH_4OH dropwise until the solution was complete. A small amount of impurities was filtered off and on acidifying the solution with acetic acid a precipitate of wedge-shaped plates[8] was obtained. Analyses on the material were as follows:

	C	H	S
Calc.	34.59	5.81	15.41
Found	34.09	5.70	15.30

III. RESULTS AND DISCUSSION

The effects of the treatments with alkali by the flow method are shown in Table 1. Confirming earlier findings, it is seen that there is good agreement between the experimental values for the cystine contents of the treated wools and those calculated on the assumption that a molecule of cystine is destroyed for each atom of sulfur lost. That little, if any, free cysteine is present in the alkali-treated wools is indicated by the values for the samples alkylated with benzyl chloride. This follows from the fact that the Sullivan method determines both cystine and cysteine but not thioethers such as would be derived from the reaction of cysteine with benzyl chloride. Had any appreciable amounts of cysteine been present, the cystine values for those samples treated with benzyl chloride would have been much lower.

The question as to the fate of the residual non-cystine sulfur in the treated wool arises. Earlier[2] it was suggested that a stable thio-compound was formed. Thus, for example, if the Schöberl mechanism were correct, a thioacetal could be formed. On the other hand, if lanthionine is the end-product, the original mechanism postulated by Schöberl would not hold. As pointed out previously, however, the reported yields of lanthionine have been so low that there would be serious objections to the assumption that the alkali-degradation of the cystine in wool resulted principally in the formation of lanthionine as an end-product.

In the present investigation, considerably higher yields of lanthionine were obtained. From a 94.7 g sample of wool that had been treated for 600 hours at 0° C, 1.47 g of lanthionine was obtained. Since as seen from Table 1, the cystine content of this sample had decreased 6.9 per cent, a theoretical yield of 5.67 g of lanthionine might have been expected. The actual yield was thus 25.5 per cent. Similarly, 2.2 g of theoretical 7.7 g of lanthionine (28.8 per cent yield) was obtained from the sample treated for 8 hours at 50° C. While these results account only for somewhat more than one-fourth of the sulfur which is no longer present as cystine, nevertheless, the amount is appreciable and in view of the difficulties involved in the isolation technique, serious consideration must be given to the possibility than lanthionine may be a principal end-product in the reaction of the cystine in wool with alkali.*

It should be pointed out that lanthionine might conceivably be formed by the reaction of cysteine with a dehydroanaline residue which might be formed from the reaction of the serine in wool with alkali[9]. That this was not the case is indicated by the data in the last column of Table 1, which show that at 0° C. the original content of serine has not been altered by the alkali treatment.

If, as the results of the present investigation indicate, an appreciable amount of sulfhydryl groups is not present in the alkali-treated wool, there still remains to be explained the apparent abnormal base-binding capacity of wool. It will be recalled that wool fails to reach equilibrium with alkaline solutions in titration experiments, a fact that was attributed[14] to the reaction of the base with the disulfide groups of the cystine. More specifically, on the assumption that the cleavage proceeds according to the Schöberl hypothesis, it was postulated that the "extra" base was bound by sulfhydryl groups (eq. 1). Obviously, this postulate is no longer valid and a new explanation

* This suggests that the permanent set, of the type produced by treating wool fibers with alkali, may result in part from the formation of lanthionine. Such a possibility has already been pointed out by Speakman and Whewell[3], since they have suggested that -C-S-C- bonds might be formed under these conditions.

must be sought. The earlier titration experiments indicated that the apparent "extra" base bound was equivalent to the sulfur which split from the wool. In light of Nicolet's hypothesis (eq. 4, 5, 6), the phenomena could be accounted for if the sulfur which is split from the wool (eq. 5) reacted with base in such a way as to consume one equivalent of base per sulfur atom. In order to test this possibility, 0.0109 g of sulfur was dissolved in 20 ml. of ethyl alcohol* and the solution mixed with 20 ml of 0.1242 *N* sodium hydroxide. The solution was kept at 25° C. and aliquots withdrawn at intervals, and titrated with standard hydrochloric acid. The results are given in Table 2.

TABLE II

Reaction of Sodium Hydroxide with Sulfur (0.0109 g.) at 25° C.

Time	*Total NaOH present M-eq.*	*NaOH consumed M-eq.*	*M-eq. NaOH consumed per M-eq. sulfur*
0	2.48	..	..
1	2.32	0.16	0.50
20	2.19	.29	.87
48	2.18	.30	.89
115	2.18	.30	.89

It will be seen that nearly one equivalent of sodium hydroxide per atom of sulfur has been consumed. While the nature of reaction of sulfur with sodium hydroxide is not clear, nevertheless the results are sufficiently close as to be able to account for the so-called "extra" base take-up in titration experiments. Finally, it may be pointed out that the results of these titration experiments, although constituting only indirect evidence, may be considered as further support for the mechanism proposed by Nicolet and Shinn.

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* Sulfur is not readily soluble in dilute NaOH. In the degradation of the cystine in wool, atomic sulfur is involved and would immediately go into solution.

HEATING EXHAUST STEAM

W. F. SCHAPHORST

HAVE you ever noticed that in the summer time it takes less gas to heat your bath water than in winter? Or, if your bath water is always warm but at times not quite warm enough for a bath, have you noticed how little gas it takes to bring the temperature up to where you want it?

That is an important fact that is too frequently overlooked by plant operators and hard-headed business men. That fact contains a worth-while lesson. Most plant operators know that exhaust steam, for instance, contains heat. But many of them do not know that exhaust steam contains the major portion of the heat that was originally obtained from the fuel. Because of this high heat content exhaust steam should always be used in one way or another, if possible.

Steam exhausted into the atmosphere has a temperature of only 212° F. When that temperature is not high enough to do a certain heating job for you, remember the above bath tub experiences. By simply adding some heat to the exhaust you can attain almost any desired temperature. The exhaust is simply run through a superheater or heat exchanger and out it comes—just as hot as needed for your purpose.

A manufacturer who wanted to maintain a temperature of 250° F. in a drying room had been in the habit of exhausting the steam from his simple engine into the atmosphere where it went to waste. He had been using live steam for the purpose because he had been led to believe that live steam was least expensive and that it was the best medium.

But he has learned a valuable lesson and now he does things differently. He just adds 38° F. to the exhaust steam by passing it through a heat exchanger in the furnace of his boiler, and the required 250° F. is easily attained.

Where exhaust steam can be used in this way it is plain that a condensing engine or a condensing steam turbine would be of considerably less value than the inexpensive, simple, non-condensing engine. It is impossible for a condensing engine or turbine to utilize even 40 per cent of the heat of the steam passing through it. The highest efficiency this writer knows of in steam plants was reported by the Twin Branch of the Indiana and Michigan Electric Co. in 1941. That plant reported an over-all thermal efficiency of 33.49 per cent. That is very good, and perhaps the writer should not say that 40 per cent is "impossible" because, some day, it may be attained.

However, what the writer is driving at here is that a simple engine can, by following the method outlined above, utilize nearly 100 per cent of the heat in the fuel by turning the exhaust to some useful purpose in heating coils, heating rooms, drying rooms, and other processes requiring a steady heat, but, in which a steam pressure is not so important. If a higher pressure is needed that, too, can be done—by means of a booster. The important point today is: Don't waste heat.

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MARCH 29, 1943

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Chemical Finishes for RAYON FABRICS*

DR. HUGH H. MOSHER

Director Textile Research—Onyx Oil & Chemical Co.

MILITARY researches have demonstrated the merits of chemical finishes in enhancing the utility and serviceability of textile materials. Sized parachute cloth has shown greater resistance to tearing and wear; waterproofed fabrics have produced fewer laundering problems and certain resin finishes have not only extended the life of a material but, when cut into garments, have supplied the user with greater warmth in winter and, paradoxically, with greater comfort in summer. The use of mildew, moth and bacteria proofing agents not only have protected the treated materials from decay, but have acted as an aid in protecting the user from the discomforts of pests and vermin and the spread of disease.

These and other benefits of textile sizes have been widely disseminated by the press and by popular publications and as a result both the manufacturer and the buying public have become finish conscious and less inclined to consider a chemical size merely as a transitory artifice designed to mask some structural weakness than as a merchandising asset.

FINISHING OBJECTIVES

All textile fibers and fabrics possess certain inherent weaknesses. These may be due to adverse characteristics in the component filaments or to some fault in the constructural design of either the yarn or the materials derived from it. Many of these adverse properties can be masked or rendered less conspicuous by the application of suitable film forming sizes. In the past the finishing of fabrics was largely confined to the employment of such films to textiles. It was also discovered that such finishes produced better drapability to garments, a new appearance and also permitted manufacturers to similate with inferior materials the hand and appearance of more costly merchandise.

Such finishing objects were of doubtful benefit to the ultimate consumer. The finishes were transient in character and the film masquerade detected after a single laundering thus engendering ill will and frequently discrediting finishing sizes.

This type of processing seems destined to play an increasingly minor role in the textile industry and is being gradually replaced by chemicals and sizes which can successfully obviate inherent textile faults or confer to fibers or fabrics new properties and characteristics to meet some popular demand for enhanced charm or utility.

*Presented at meeting, New York Section, March 5, 1943.

INHERENT RAYON DEFECTS

Rayon yarns possess visual, tactile and structural weaknesses. The fibers are often too bright and lustrous, possess a rather cold harsh hand and may even lack the requisite strength and elasticity, particularly when wet, to give satisfactory service for some desired purpose. The smooth peripheries of the yarns also cause the threads to slip on each other in sheer fabric constructions and as a consequence such fabrics are inclined to exhibit non-uniform spacings in apparel.

In common with all continuous filament yarns, rayon fibers tend to chafe, scuff and fray in use, and to exhibit the universal cellulosic weakness of shrinking and crushing easily. The rayons as a class are rather hygroscopic, but even this property may not be optimal, and it may act as either an asset or fault depending upon the purpose for which the fiber is to be used.

Rayon also shares the universal textile defect of being combustible, and is easily attacked and weakened by certain moulds, bacteria and certain insect pests.

It is for the correction of these and other defects that rayon finishes are applied, but it is not alone in the correction of rayon defects that finishes possess merit. Rayon is a chemically active material and by the use of certain chemicals, rayon can be made to acquire new and often unexpected properties.

SOFTENING OF RAYONS

In the field of fine fabrics both charm of appearance and lure of touch are desired characteristics. Rayon, compared with silk, is somewhat deficient in both. Its fibers lack that warm scroopy softness and suppleness of the silk worms product, but certain finishing agents have the property of minimizing the difference.

Sulfated oils and esters were for years the accepted standard softening agents and even today advanced types are unsurpassed for certain types of rayon materials particularly when applied with other auxiliaries.

With advances in the art of finishing and the development of new synthetics, emulsified lanolins and lecithins and their esters came into use and these in turn were supplanted by partially sulfated fat alcohols and other primary and secondary alkyl amide phosphates and sulfates.

The fatty alcohols proved of particular interest. It was found that by altering the methods of preparation that they could act either as a potent softening agent without scroop-

ing action or similate on rayon that soft alluring crunch that for years rendered silk unique among fibers.

The most outstanding advance in the softening of rayons, however, did not occur until the cationic softening agents were synthesized, and found application in the textile industry.

These compounds, in contradistinction to the older types of agents, bear a positive electrical charge. This charge, on suitably formulated softening agents, permits a semi permanent union between the agent and the treated fiber and produces such a superiority of plasticizing action and surface orientation, as to yield a vastly superior softening effect.

The use of the older types of cationic softening auxiliaries were not without certain hazards. The agents tended to yellow "whites," changed the caste and hue of certain dyestuffs and reduced the light fastness of applied vat and direct colors, but in spite of these defects the better balanced cationic softening auxiliaries were such superior softening agents that their short comings were tolerated.

Science rarely permits her defective materials to survive and in keeping with her usual practice the defects were first traced to factors in ionization; these were restricted, and the so-called cationic softening agent became almost universally accepted as the standard of advanced finishing, except where properties other than softening were required.

It must be added, that the true cationic softening agents are inimical to anionic sizes and the two can seldom be employed from the same finishing bath.

It is not solely as a softening agent that cationic auxiliaries receive employment in textile finishing. Certain representatives possess dye-setting, mildew proofing, and bactericidal properties and are even of service in insolubilizing certain resin and gum types of finish.

A very unique dulling agent has also been produced by reacting long chained cationic and anionic auxiliaries. The product of the reaction is insoluble in water and is receiving employment, as an organic pigment, in dulling certain classes of rayon.

Its delustering potencies is not particularly high and its chief claim to recognition lies in the superiority of the hand that it imparts to rayon.

DULLING AGENTS

A sheen on textile fibers and fabrics has always been held in high esteem as it imparts a striking luxurious effect. A surfeit of luster, however, may be decidedly objectionable. Excessive brilliance is too blatant, too gaudy and too inclined to act as a badge of course and vulgar tastes.

The lustrous types of rayon are inclined to exhibit excessive brilliance especially when employed in the form of loosely twisted yarns. As a corrective manufacturers have developed various qualities of dull yarn.

Such yarns owe their dullness to the presence of various pigments, or oils which had been incorporated into the rayon precursory matrices prior to filament formation. These extraneous substances modify the light reflection and thereby produce dullness.

Unfortunately such yarns possess lower tenacity and diminished elasticity and elongation, and because of these deficiencies and factors of commercial expediency, it is often desirable to employ bright yarns and restrict luster by finishes.

Any agent which modifies the reflection of light by rayon acts as a duller.

Many types of delustering agents have been proposed for this purpose. Surface etching of the yarns by the use of thiocyanates and other chemicals have been employed; metallic weighting agents, notably insoluble calcium stannates, have been found extremely effective, but such methods are costly, difficult to apply, tend to seriously weaken the rayon and produce an objectionable hand. As a result the pigment types of dullers are almost universally used.

In the production of rayon dulling agents manufacturers and scientists have appropriated pages from the paint makers manuals. It has been found that those white pigments with greatest spreading power possess the most potent dulling action on rayon fibers. However, the lesson gained from the paint industry requires intelligent application. The toxicity and dermatological influence of pigments on the human system is of far less importance in paints than on materials that may be constantly handled or worn in contact with the human skin such as a textile. Moreover, the pigments must be capable of emulsification in aqueous solutions.

After considering all the factors involved, the number of pigments available for dulling is not large. Titanium dioxide, barium sulfate, zinc oxide and various types of clays and talc are about the only natural pigments suitable for use. Of these titanium dioxide is by far the best.

Its pigments disperse readily through the mediation of a large variety of emulsifying agents. However, the dulling potencies of the emulsions is not identical and show great variations in effectiveness. All of the factors influencing dullness have not as yet been determined but it is certain that particle size, penetration and surface orientation are determining influences.

But titanium dioxide is by no means a dulling panacea. In itself it is inclined to impart a dry hand to rayons, accelerates the fading of certain direct colors by sun light and produces a frosty over caste to fabrics dyed dark-shades.

These inherent defects have, in part, been corrected. The envelopment of the dulling particle in a film of wax, fat or better, cationic softener, eliminates much of the dryness of touch that it produces on textiles and the fading tendency of titanium dioxide on direct colors can be

inhibited by various waxes and chemicals. Chemists have not as yet been successful in eliminating the white caste on dark color shades and as a result "titanium" dullers are not commonly used on rayon fabrics dyed dark browns, blue or blacks.

Complete fabric dullness is not always a rayon finishing pre-requisite and where a mere mellowing of excessive gloss is demanded the finisher has at his disposal a host of natural and synthetic waxes, resins, and waterproofing agents. A poly functional finishing auxiliary is usually employed where semi-dulling is required, an agent which will not only inhibit the luster of rayon but impart some other desired characteristics such as water-resistance.

RAYON SIZING AGENTS

Although rayon fabrics possess structural weaknesses, particularly when wet, these are capable of being masked by corrective finishes. The rayons, however, are not as commonly sized as other textile materials.

Their yarns are inclined to be cold and harsh and most corrective finishes accentuate these properties. Many also embrittle the fabrics, cause them to tear readily and give a false impression of weakness. As a consequence the sizing of rayon fabrics is most advantageously accomplished by the use of soft, plastic resins and waxes such as latex, the alkyd and similar resins, waterproofing agents and various pliable synthetics.

This does not imply that for special effects, film forming materials may not be advantageously used.

Probably the most commonly applied rayon sizing agents have been the urea-formaldehyde types of resin. They may be classified as poly functional finishing agents which term correctly implies that they are useful in accomplishing any of a number of diverse objectives—usually corrective.

Among the inherent weaknesses of rayon fabric must be included their tendencies to crush and wrinkle, their inclinations to stretch in use (this applies particularly to materials derived from spun yarns) and shrink during laundering and the ease with which their fibers slip in sheer constructions producing dense and sparse areas.

These potential defects have in the past restricted the use of rayons in certain textile fields.

The urea-formaldehydes have proved an excellent correcting agent for the faults enumerated. Its presence confers to many types of rayon materials crush and crease resistance; it regulates the stretch and shrinkage of fabrics composed of spun rayon yarns and to a lesser degree to those prepared from continuous filament and it inhibits the slippage of the yarns in sheer fabrics.

The successful application of the resin to fabrics is more complex than the usual textile finishing operation. Adherence to details must be practiced and deviations exact painful retributions in weakened merchandise and in the development of obnoxious odors.

Although urea-formaldehyde complexes have been of inestimable value to the rayon industry they are not panaceas. There has been a tendency to exaggerate their merits and minimize their defects.

Urea-formaldehyde resins are obtainable in several states of polymerization. The less highly condensed forms are water soluble and largely used in producing crush-proof effects and in rendering materials "shrink-proof." The resins are applied to fabrics and frequently affixed by catalysts, usually acidic, at elevated temperatures. Comparatively high concentrations are required to secure a crush-proofing effect. Almost every type of rayon construction shows a different response to the crease-proofing action of the resin. Spun rayon constructions are particularly susceptible whereas certain continuous filament yarn fabrics do not respond perceptibly.

Where complete resin permanence is required the materials should be washed in dilute soap or synthetic detergent solutions. Lower concentrations of the resins act as aids in regulating the stretch and shrinkage of certain classes of rayon fabrics such as those composed in whole or part of spun yarns. Regardless of the objective it is essential that the resin application be regulated and controlled so as to avoid the development of obnoxious odors during aging.

Although crush-proofed fabrics have been reputed to possess greater tenacity than untreated materials, it is the writer's experience that a certain loss can hardly be avoided. Crush-proofed fabrics likewise are sensitized to abrasive agencies. However, the benefits derived are ordinarily sufficient to more than compensate for any theoretical loss in wearing service. Where lesser amounts of resin are applied, as in rendering fabrics shrink resistant, slight strength increases may be obtained in certain cases.

The more highly condensed resins are valuable rayon sizing materials, act as lustering agents for taffetas and are applied to render fabrics slip-proof.

The ease with which the component yarns of a rayon fabric slip may be attributed to the smooth peripheries of the fibers. Any agent which will cement the fibers together at their points of juncture will tend to maintain the yarns in set positions in a fabric. Properly condensed urea-formaldehydes are serviceable for this purpose though inferior to many of the natural and synthetic wax and resins complexes.

All fabrics possess characteristic physical and chemical properties. These frequently preclude their use for some designed objective, but when the inherent characteristics are modified or changed to mask some adverse property the fabric may be said to have been proofed.

Thus rayon may be rendered non adsorbent to water by the application of aluminum wax complexes, oil proofed by various waxes and resins and fire proofed by various ammonium salts, borates, chlorinated hydrocarbons or antimony oxide.

The proofing of fabrics for some designed objective constitutes an important function of finishing.

(Concluded on page P156)

DYEING OF RAYON

and Some New Developments*

JAMES HOPWOOD
American Viscose Company

I. HOSIERY

a. Full Fashioned Viscose

WE ARE doing very few hose containing combinations of fibers, mostly all viscose and in any case you all have your dyeing formulae set for viscose and cotton and know how to achieve a union. Therefore, I will give you our method of handling. We use the single bath method, entering the hose at the boil into 6 per cent soap—2 per cent Pine Oil and 3 per cent TSPP. Run for ten minutes then add Glauber's Salt in small portions—no more than 5 per cent in all.

b. Full Fashioned Acetate

The pre-boarding machine is set at 180° F. and after the door closes the temperature increases to 206° F. The stockings are left in for three minutes. Hose are taken wet from the machine and hung by the toes from a clip in a drying cabinet until dry. Scour for ten minutes at 160° F, dye at 160 to 165 and cool slowly after both scouring and dyeing. This is very essential. Finish, with minor variations, the same as for viscose. Do not pack more than one layer of bags of hose in boxes when standing or in the extractor. Board at 170 to 180° F.

c. Seamless Rayon Hosiery

1. *Scouring*

One of the major causes for dyeing difficulties on rayon hosiery is the incomplete removal of oils, waxes, solvents and other materials used in preparing the yarn for both winding and knitting. While this appears to be a matter of common sense, it is frequently overlooked, as the formula and treatment will often have to be modified to meet varying conditions both as to kinds of material to be removed and other variables due to storage condition of greige hose.

In the case of hosiery stocks which have been stored for some time in the greige before scouring, the oils and various sizing compounds can oxidize even to the point of acquiring varnish-like characteristics which render them exceedingly difficult of removal. Wherever possible, scouring should occur as soon as possible after knitting, but under some market conditions where color selection is delayed, it is often wise to scour out the goods separately and hold them in the boiled-off state until ready for dyeing.

Members of our technical staff have in more than one instance checked the temperatures of unscoured hosiery in storage bins and found through chemical action that actual heating developed deep in the bins within the stockings. Such a condition could not only weaken the hose to the point of producing excessive break outs during boarding but also would render the sizing so difficult of removal as to cause streaky dyeing. Oxidation of the various sizing products on rayon yarns can produce oxycellulose, which is not only non-hydroscopic but which is also dye resistant. In addition, it is woefully lacking in strength. For these reasons, it is imperative that scouring formulas be so adjusted and the scouring operation be done within a reasonable time after knitting so as to avoid such difficulties. These principles apply to rayon in the knitting and textile fields in general rather than to the hosiery field alone.

Under favorable conditions, scouring and dyeing of hosiery can be conducted in a single bath but whenever there is any question as to conditions of storage and difficulty of size removal, it is wise to scour separately to insure complete boil-off.

It is also probably stating the obvious to say that it is essential to have good clean soft water for use in the scouring operation, particularly in the presence of such detergents as soap, but we feel this fact worth mentioning as it is one that is overlooked in checking back as to cause for dyeing difficulties.

The complete scouring of rayon hosiery is rendered more difficult today than it was a short time ago because of the use of high twist yarns and the resulting greater difficulties of bath penetration.

2. *Dyeing*

Several years ago the orthodox method of dyeing hose was to enter the greige goods at low temperatures and to bring the dye bath up slowly to the boil with the idea that this was necessary and essential for leveling dye results. This method was also general practice on silk hosiery. In considering dyeing procedures for rayon, members of our technical staff have worked from theory to practice along the lines of the reaction of cellulosic products to temperature. The theory was developed that at high temperatures the minute openings in the molecular structure of the cellulose are expanded because of the swelling of the

* Presented at meeting, Mid-West Section, February 6, 1943.

cellulose. Under such conditions it was reasoned that the faster dyestuffs with their higher molecular size and weight could more readily enter within the cellulose at higher temperatures than at lower temperatures. This theory was put into practice on a laboratory basis and found to give promising results. It was then put to actual test in a case where one of our large hosiery customers had extensive difficulties with tiger stripes. Although it was extremely unorthodox at the time, the dyer at this mill agreed to run volume tests, entering the greige goods at the boil and also entering the dyestuffs at the boil. The results were highly gratifying in that the tiger stripes were completely eliminated in this instance and the method proved so satisfactory that it was adopted as standard. It was also found in this test case that entering the greige goods at the boil, while it offers some difficulties in handling, gave a very marked improvement in clarity and uniformity of stitch formation.

After running by this method for some time, this mill adopted the same procedure for silk hosiery also. In this instance also, the salting was found to be important being added in three stages in 10 or 15-minute intervals until bath is exhausted. There are, of course, exceptions to this general principle because of the differences in rate of absorption. In some combination dyestuffs, the principle is offered as a matter of interest for proof in individual cases and under individual local conditions.

II. THE DYEING AND FINISHING OF WARP KNIT FABRICS (Tricot)

In our development work on warp knit fabrics at our Textile Unit we have found it to be of decided advantage to handle this fabric in tubular form rather than in flat goods. During the knitting operation we use a marking device at the selvedges of the fabric, which device automatically stamps at regular intervals these selvedges to insure proper fabric alignment during the seaming into tubular form. In the seaming operation we have found it to be of greater help to equip the machine with a pair of reverse scrolls which act as an uncurling device to greatly facilitate this seaming operation.

Our method of wet processing Acetate Tricot consists of wetting out the tubed fabric in as close to full tubular width as possible on an endless blanket upon which a hot soap solution is sprayed. From this machine one piece at a time is loaded into the vessel and stranded up for scouring and dyeing.

Dyeing temperature does not exceed 165° F. and bath is cooled very gradually after shade is reached.

During an earlier study of this industry we found that one of the basic problems had to do with fabric stability in the finished garment. In order to help in the solution of this problem we sent a member of our staff to Europe just prior to the outbreak of World War No. 2. Methods of finishing tricot fabrics were studied both in England and on the Continent, and in Germany a drying and finishing machine was uncovered which used a principle radically different from that in common use here. This principle involved the pulling or drawing of hot air directly through the knit fabric instead of glancing off the surface as it does on the well known festoon or conveyor types of dryers. It was also discovered that some ten years ago one of these machines had been delivered to this country and after somewhat of a search this original machine was located in a warehouse in Maine. It had been discarded for commercial use because it had been unsatisfactory for the purpose intended, namely, its use on woven piece goods.

This machine was acquired through the co-operation of Proctor and Schwartz and brought to the Textile Unit to perfect its use for warp knit fabrics. Special spreaders were designed and improvements in general were made to bring it into successful operating condition. By means of an over-feeding device longitudinal fabric stretch was controlled; by means of the spreading mechanism and a fabric blanket, lateral fabric stretch was controlled. The net result was a machine which not only gave excellent results on fabric stability but a means of greatly reducing cost of drying and finishing of this type fabric. Since the perfecting of this original model the machine has been turned over to Proctor & Schwartz, whose engineering staff worked with ours throughout this development. They have in turn built the first large scale machine with a suction drum with approximately 50 per cent larger diameter and twice the axial length of the original cylinder. This machine is now in practical operation in a mill and only the shortage of critical materials has delayed the construction of additional machines. Naturally and obviously this system of drying and finishing lends itself readily to circular knit fabrics.

III. WOVEN FABRICS

a. The Scouring of Viscose Fabrics Spun and Filament

In general preparation consists of the following:

For starch sized goods an enzyme treatment is given.

For gelatin sized goods either an enzyme treatment or cold water soaking are given to swell the size and facilitate its subsequent removal.

A popular initial treatment for pigment rayon taffeta is a cold open width 7° Twaddle caustic impregnation. The boil off proper may consist of a scour with soap, or one of the many sulfonated fatty alcohols with TSPP. This is usually carried on at the boil. Bleaching is given when necessary. Viscose woven fabrics are prepared for dye either open width or in the rope form or by a combination of the two.

b. Open Width

This takes place on the jig or on a continuous machine consisting of a number of separate compartments with squeeze rolls in between. Such fabrics as satins, taffetas, twills and serges may be run on the above as they are not constructed so as to be run in the rope form without cracking. For crepe and pigment taffeta, many open width

scouring machines are available, each of which have this in common—that the cloth is evenly and thoroughly wet out before wrinkles have a chance to develop and that the fabric is run with a minimum of tension.

c. Rope Form

This is carried out in the beck or vessel or continuous rope scouring machine but after first thoroughly wetting out in the open width. Unless this is done cracks or rope marks will develop. For certain fabrics which require special care in handling it may be necessary to stick boil off. This consists of reeling the cloth in the hank form and suspending on smooth sticks or bent metal rods in the boil off liquor, usually in a wooden tank. The hanks are turned manually until preparation is complete and are then ready to be dyed in rope form. Certain special fabrics are hanked or folded and suspended in the boil off liquor from strings stitched into the selvedge.

d. Dyeing

Viscose flat goods or fabrics for open width dyeing such as satins, taffetas, serges, twills, etc., are dyed either on the jig alone or by padding through color to gain penetration, then giving ends on the jig through dye liquor to complete the dyeing. Acetate in similar fabrics is straight jig dyed.

Vat colors are applied most successfully by the pad and jig method. Both the pigment pad and the reduced pad method have their adherents. On spun fabrics yarn tension should be kept as low as possible at jig reduction and oxidation should be followed by a rope soaping whenever the fabric permits to give the desirable woolly hand.

Dyeing crepes or spuns in the rope form is carried out on oval reel vessels. After scouring in the continuous machine the dyeing bath is made up with soap, TSPP and a penetrant. The liquor level should be higher for heavy goods than for light fabrics to obviate chafe and rope marks. Always remember that at the end of the dyeing operation the dye bath should be cooled down very gradually to avoid cracks. If it is wished, finish can be applied in a fresh bath.

e. Finishing

Dress fabrics, crepes or spun rayon, after dyeing, are given a light centrifugal extracting or in some instances an open width vacuum extracting. Drying is usually carried out in the slack dryer with as little tension as possible. Slack dryers are usually fitted with a quetsch for applying finish in the front of the machine. A softener, often cationic, is used on dress goods to give the characteristic drapey hand. Decating is an operation much used to hide the small breaks caused by rope dyeing. Fabrics which have been dyed in the open width are usually vacuum extracted and run through a finish on the quetsch, followed by can or Palmer drying in conjunction with the tenter frame. Calendering and steam frame finish the operation on these fabrics.

IV. METHODS OF COLOR SELECTION

In Whittaker & Wilcock—"Dyeing With Coal Tar Dyestuffs" there is a chapter devoted to color selection, the general tenor of which follows:

If we consider dyeing as a process of diffusion which seems to be the generally accepted theory, then an equilibrium must eventually be established between the dye on the yarn and the dye remaining in the solution. In other words, when equilibrium has been reached, there will be as much dye coming off the fiber and going into solution as there is going on to the rayon. The time required for dyes to reach this equilibrium varies greatly, the level dyeing colors reaching it in the quickest time.

Some years ago, Boulton and Reading published a paper on "Classification of Direct Dyes With Respect to the Production of Level Dyeing on Viscose Rayon." The scheme is as follows:

1. To determine the amount of salt necessary to give a 50% dye bath exhaustion when the dyeing is allowed to proceed to this 50% equilibrium ascertained by means of a colorimeter.

2. By means of a series of time dyeings with this amount of salt, the time required to give a 25% exhaustion is found, termed the "time of half-dyeing." This varies widely with different dyes and enables the dyer to choose colors suitable for compound shades. For instance, the level dyeing colors are the ones which have the lowest time of half-dyeing and the most unlevel, the highest. However, any dye will prove to be level dyeing if the dyeing is allowed to go to equilibrium which would in some instances take so long as to effect the destruction of the material being dyed. All this goes to emphasize dyeing's Golden Rule which is—"Dyes Chosen for Compound Shades Must Dye on at the Same Rate." We are following this up very closely using domestic dyestuffs and should soon have some valuable information.

Another fact which proves a very disturbing factor is salt sensitivity. Salt or sodium sulfate used in standardization of dyes is sufficient to give bad results and even the natural salt content of the water used has given trouble. Without question, the addition of salt to the dye bath should be carefully controlled and not dumped in wholesale as so often occurs.

GENERAL DEVELOPMENTS IN RAYON INDUSTRY

Rayon as we know it today is a cellulose product and a vastly better one than the coarse fiber of the early days. I have been asked to dwell for a time on new developments in the industry and possibly the high tenacity yarns should be first on the list. The Tenasco yarns manufactured in this country by American Viscose are types of these modern high strength yarns. In the field of heavy duty tire fabrics, this type yarn has come to the fore. Extensive experiments have shown that it is very resistant

to sudden shocks and has more elasticity than ordinary rayon and cotton. Neither does it get as hot as cotton fabric in tires. Such yarns with a dry strength of 3.6 grams per denier and 9% extensibility, 2.2 grams per denier wet strength and 15% extensibility compared with 2 grams per denier dry and 19% extensibility and 1 gram wet with 22% extensibility—the wet strength of "Tenasco" is seen to be greater than the dry strength of ordinary rayon. 3.6 grams per denier corresponds to approximately 70,000 pounds per square inch, the strength of a fairly good steel. "Tenasco" will be a definite factor in the hosiery of the future. None is now available for civilian use, the yarn being used at present for war orders. Our research men promise a yarn suitable for hosiery after the war and comparatively low in cost.

One of the most striking developments of recent years is the tremendous growth of rayon staple developed in Germany and Italy as substitutes for wool and cotton. This was not at first of interest to us in the United States. However, its good points became apparent not as a substitute but as a means of obtaining variety in fiber design. The fiber is exactly the same as continuous filament yarn which is cut to suitable lengths for use on cotton, wool and worsted machinery.

Another significant development has been produced by Courtaulds in England and known as Rayolanda. This product is dyed by both wool dyes and direct colors and is a basified fiber which means that it is a cellulose fiber having a basic nitrogenous material added, using the cellulose molecule as a framework for holding the basic constituents which act as the dye absorbers. Rayolanda has a soft, warm, wooly feel and has greater affinity for direct dye than rayon. Because of this great affinity, cross-dyed and two-tones effects can be obtained with spun rayon and Rayolanda. Rayolanda has slightly lower tensile strength than straight "Fibro" but due to the treatment received to make it wool dyeing is more water repellent and so dries more readily. Fastness to washing of direct colors on this fiber is excellent, but colors are less light fast.

Vinyon

Vinyon, which has somewhat similar properties to nylon, is a co-polymer of vinyl chloride and vinyl acetate. It is thermoplastic at temperatures over 150° F. and at this temperature shrinkage takes place. After spinning, the yarn is stretched to twice its original length. This plays a large part in "Vinyon's" high strength which may be controlled between 1 to 4 grams per denier. The strength of wet and dry yarn is the same, the yarn being extremely water repellent (less than 1% water retention). It is used or proposed for use, in the following fields: filter fabrics, acid and alkali resistant clothing, industrial uses, waterproof clothing, electric insulation, shower curtains, awnings, hosiery, and mesh shoe fabrics. Until recently, when production was frozen, fair-sized quantities of fabrics were dyed and thermally set for use in the women's shoe trade. The dyeing has been accomplished by use of Calco's Calcovin colors or the alternative method of using acetate colors with Dowicide No. 1, keeping the temperature below 150° F.

Chemical Finishes—

(Concluded from page P152)

Our military bureaus have demonstrated the practical merits of textile finishing. The public has become finish conscious and receptive, as never before, to any method of processing which will enhance the lure and utility of rayons.

New synthetics and resins are the tools to evolve rayon into objects of unexpected utility and charm but the textile chemist must still act as guide and craftsmen.

MEETING, PIEDMONT SECTION

THE first 1943 meeting of the Piedmont Section was held in Greenville, South Carolina at the Poinsett Hotel March 6th.

Lieut. Colonel Thomas D. Lewis, officer in charge of Duck, Webbing, and Thread of the Procurement Division, Jeffersonville Quartermaster Depot, discussed, "The Overall Picture of Dyeing and Finishing of Such Materials As They Concern the Jeffersonville, Indiana, Quartermaster Depot."

Elmer C. Bertolet, Senior Technologist of the Engineering Division of the Jeffersonville Depot discussed in a most comprehensive manner, "The Finishing of Army Ducks with Particular Reference to Mildew Proofing."

Approximately 240 members and guests attended the dinner which was served at 7:30 p.m. C. Norris Rabold, chairman and chemist for the Union Bleachery, presided at the meeting. T. R. Smith, National President and P. J. Wood, past National President were among the guests at the head table.

Members from the following sections were also welcomed guests of the Piedmont Section—Northern New England, Rhode Island, New York, Philadelphia, South Central and Southeastern sections.

The Southeastern section with 12 visiting members had the largest delegation of guests. The Piedmont Section is always happy to extend their hospitality to visiting members and friends.

The next meeting of the Piedmont Section will be held in Greensboro, North Carolina on June 5th—Charlotte, North Carolina on October 30th (tentative date).

MEMBERSHIP APPLICATIONS

Corporate

Arnold-Hoffman & Co., Belding Heminway Co., Commonwealth Color & Chemical Co., Southern Dyestuff Corporation, Wyandotte Worsted Co.

Senior

Talcott D. Andrews—Laboratory, Hellwig Silk Dyeing Co., Philadelphia, Pa. Sponsors: A. W. Etchells, S. Aumuller.

William Samuel Armitt—Secretary-Treasurer, Narrow Ribbon Specialty Mills, Inc., Hanover, Pa. Sponsors: H. B. Kime, W. O. Neeb.

Henry D. Barker—Principal Pathologist, Bur. of Plant Industry, U. S. Dept. of Agriculture, Washington, D. C. Sponsors: T. R. Smith, W. G. Chace.

John P. Bauer—Sales Manager, Ciba Co., Inc., New York, N. Y. Sponsors: R. Fitzgibbon, T. F. O'Brien.

John Carey Beard—Overseer of Finishing, Erwin Cotton Mills Co., Erwin, N. C. Sponsors: H. Puckett, Q. M. Rhodes.

James F. Best—Junior Chemist, National Aniline Div., Allied Chemical & Dye Corp., New York, N. Y. Sponsors: J. E. Loughlin, W. A. Holst, Jr.

Max M. Berliner—Foreman, Loebl Dye Works, Inc., Roanoke, Va. Sponsors: H. L. Pearson, J. E. Loughlin.

Brindley Jack Brown—Research & Development Executive, Thomas French & Sons, Ltd., Manchester, England. Sponsors: E. J. Monaghan, C. F. Schaumann.

James P. Catalino—Foreman, Bleach Dept., Delta Finishing Co., Philadelphia, Pa. Sponsors: J. P. Conway, R. M. Ritter.

Joseph H. Condon—Finisher, R. Wolfenden & Sons, Attleboro, Mass. Sponsors: R. W. Joerger, H. V. King.

Aubrey Douglas Dow—Dyer, American Woolen Co., Brown Mills, Dover-Foxcroft, Maine. Sponsors: A. Morrison, R. E. Noyes.

Louis W. Fall—Associate Commodity Standards Specialist, Standards Div., O.P.A., Washington, D. C. Sponsors: H. S. Schenker, M. L. Chandross.

William F. Ferrazano—Salesman, National Aniline Div., Allied Chemical & Dye Corp., New York, N. Y. Sponsors: C. C. Knights, R. F. Holoch.

Arthur L. Fox—Application Research, General Aniline & Film Corp., Easton, Pa. Sponsors: C. Z. Draves, H. E. Hager.

Robert K. Fox—Chemist and Partner, The Fox Chemical Co., Coshocton, Ohio. Sponsors: J. R. Redmond, E. C. Bertolet.

H. Martin Friedman—Research Chemist, Hart Products Corp., New York, N. Y. Sponsors: S. M. Edelstein, R. Hart.

Bert Gilbert—Supt., Yarn Dept., Lawrence Woolen Co., Lawrence, Mass. Sponsors: L. A. Olney, B. A. Ryberg.

George J. Greendonner, Jr.—Colorist, Phoenix Color & Chemical Co., Paterson, N. J. Sponsors: C. L. Daley, H. C. Chapin.

William J. Greene Jr.—Supt., Sumter Textile Mills, Sumter, S. C. Sponsors: E. T. Mobley, D. B. Osborne.

Ira Lee Griffin—Southern Manager, Stein Hall & Co., Inc., Charlotte, N. C. Sponsors: D. E. Truax, W. S. Gilbert.

William Joseph Hawk—Dyer, Belding Hosiery Mills, Chicago, Ill. Sponsors: L. Hoehn Jr., B. H. Sharman.

Arthur J. Heinzmann—Overseer of Finishing, Layout and Scouring, Lawrence Print Works, Lawrence, Mass. Sponsors: H. E. Wieland, J. N. Dalton.

Palmer G. Hendrix—Demonstrator, Specialty Products Co., Jersey City, N. J. Sponsors: A. Rotcop, L. P. Brick.

Charles H. Henkels—Asst. Textile Research Engineer, Talon, Inc., Meadville, Pa. Sponsors: G. B. Harvey, E. C. Bertolet.

Carl T. Herzfeld—Director of Quality Control, Chicago Mail Order Co., Chicago, Ill. Sponsors: A. G. Alexander, A. J. Olson.

Robert E. Horsey—Specialty Products Div., Tech. Sales, Givaudan-Delawanna, Inc., New York, N. Y. Sponsors: E. Freedman, M. Schuster.

William A. Hughes—Capt., C.W.S., Niagara Falls Plant, Niagara Falls, N. Y. Sponsors: R. Hart, S. M. Edelstein.

John L. Kurlychek—Research Chemist, U. S. Rubber Co., General Laboratories, Passaic, N. J. Sponsors: A. K. Gyzander, C. C. Knights.

Joseph C. Large—Supt. of Dyeing and Finishing, Clear Spring Worsted Mills, Doylestown, Pa. Sponsors: F. Gonnerman, E. A. Lutz.

Edward Larson—Laboratory Technician, Phoenix Hosiery Co., Milwaukee, Wis. Sponsors: A. J. Olson, A. G. Alexander.

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Josef Loebl—President, Loebl Dye Works, Roanoke, Va. Sponsors: B. C. Bond, E. Empting.

C. William Mahnken—Asst. to Director of Sales, Geigy Co., Inc. Sponsors: H. C. Spatz, T. F. O'Brien, Jr.

Frank Marino—Manager and Textile Colorist, Capital Textile Printing Co., New York, N. Y. Sponsors: K. A. Sutherland, G. H. Alpers.

J. Gilbert Mearns—Vice President and Plant Manager, Ho-Ho-Kus Bleachery, Ho-Ho-Kus, N. J. Sponsors: P. J. Kennedy, T. F. O'Brien.

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August F. Ottinger—Chief Chemist, Bemis Bros. Bag Co., St. Louis, Mo. Sponsors: W. R. Guthrie, P. V. Keyser.

Carl William Patton—Development Dept., Plastics Div., Carbide & Carbon Chemicals Corp., New York, N. Y. Sponsors: H. B. McClure, F. Stutz.

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Frank H. Schortemeyer—Leather Chemist, National Aniline Div., Allied Chemical & Dye Corp., New York, N. Y. Sponsors: J. E. Loughlin, A. K. Gyzander.

William Harold Scott—Asst. Chemist, Rock Hill Printing & Finishing Co., Rock Hill, S. C. Sponsors: S. Springer, C. Forster.

Ridgley G. Shepherd—Chemical Engineer, Inspection Dept., Associated Factory Mutual Insurance Co., Boston, Mass. Sponsors: W. F. Deady, F. H. Couchman.

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Hugo H. Todebush—Salesman & Technician, Ciba Co., Inc., New York, N. Y. Sponsors: R. Fitzgibbon, T. F. O'Brien.

Virgil Henry Waite—Director of Research, McGean Chemical Co., Cleveland, Ohio. Sponsors: J. R. Redmond, E. C. Bertolet.

William D. Waters—Foreman, Woven Fabric Dyeing & Finishing, Tubize Chatillon Corp., Hopewell, Va. Sponsors: C. Waters, E. J. Stelpstra.

Paul Lawrence Weintraub, Jr.—Lt. Comdr., U. S. Navy, Naval Clothing Depot, Brooklyn, N. Y. Sponsors: L. S. Little, R. S. Lunt.

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Lawrence E. Wilkerson—Supt. Finishing & Piece Dyeing, Atlanta Woolen Mills, Atlanta, Ga. Sponsors: L. L. Dettelbach, L. L. Baker.

Harold Will—Colorist, General Dyestuff Corp., New York, N. Y. Sponsors: C. Z. Draves, J. R. Bonnar.

Joseph P. Willan—Textile Chemist & Asst. Supt. Dyeing & Finishing, Lawrence Woolen Co., Lawrence, Mass. Sponsors: L. A. Olney, B. A. Ryberg.

Junior

Margaret M. Cooper—Asst. Professor of Chemistry, Texas State College for Women, Denton, Texas. Sponsors: P. B. Mack, C. R. Phillips.

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Raymond S. Gregson—Textile Testing, Worcester Textile Co., Inc., Greystone, R. I. Sponsors: H. V. King, F. A. Prisley.

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Jones Y. Pharr, Jr.—1st Lt., Quartermaster Corps, U. S. Army, Charlotte, N. C. Sponsors: T. R. Smith, S. M. Cone.

Associate

Karl E. Buff—Chemist, National Automotive Fibers, Inc., Detroit, Mich.

James Scott Long—Chemical Director, Devoe & Raynolds Co., Inc., Louisville, Ky.

Edgar L. Schlesinger—Converter, Cohn Hall Marx Co., New York, N. Y.

Frank K. Smith—Asst. to President, Alrose Chemical Co., Cranston, R. I.

Leonard E. Turits—President, Continental Chemical Co., Passaic, N. J.

August von Hagen—Commodity Contract Clerk, U. S. War Dept., Quartermaster Depot, Philadelphia, Pa.

Richard D. Wells—Textile Technologist, U. S. War Dept., Office of Quartermaster General, Washington, D. C.

Otto R. Wieland—Garment Dyer, Wieland's Dye Works, Methuen, Mass.

Ernest Zerner—Research Director, Warwick Chemical Co., New York, N. Y.

Student

Norman J. Alexander—Straubenmuller Textile High School. Sponsor: P. T. O'Brien.

Edwin Gottlieb—Lowell Textile Institute. Sponsor: L. A. Olney.

James E. Orr, Jr.—University of Delaware. Sponsor: A. S. Eastman.

Applications for transfer to Senior membership, Everett T. Lesure, Arthur H. Noble, Jr., James E. Turner, Jr.

APPLICATION BLANKS

for Individual

and Corporate

MEMBERSHIP

may be obtained from

the Secretary

Dr. H. C. Chapin, Lowell Textile Institute, Lowell, Mass.

• CALENDAR •
OF COMING EVENTS

Meeting, New York Section, Downtown Athletic Club, New York City, April 16, 1943. Speaker: Lt. Col. Frank M. Steadman, Philadelphia Quartermaster Depot, will discuss research and development in the Q. M. C.

General Business Meeting (in conjunction with New York Section meeting) April 16, 1943, New York City.

Meeting, Northern New England Section, April 16, 1943.

Council and Research Committee Meetings, April 17, 1943, Engineering Society Building, 29 West 39th Street, New York City.

Meeting, Southeastern Section, Atlanta Athletic Club, Atlanta, Ga., April 17, 1943.

Meeting, Philadelphia Section, April 30, 1943.

Meeting, Piedmont Section, Greensboro, N. C., June 5, 1943.

• CLIMATIC RESEARCH LABORATORY

A Climatic Research Laboratory for testing and evaluating the effectiveness of food, clothing and military equipment on human subjects under actual conditions of heat and cold has been established by the Quartermaster Corps at Lawrence, Massachusetts, the War Department announced recently. Active research and experimental work is expected to get fully under way in the near future.

The laboratory was established to determine clothing and other requirements of soldiers serving in areas of extreme climatological conditions where it is impossible to make adequate field tests because of the season of the year or lack of time in which to carry them out under field conditions. The laboratory is already in partial operation and when completed will be capable of creating any condition of temperature, humidity, wind or radiation which might be encountered anywhere on the face of the earth.

The cold chamber, around which the laboratory is being built, has been reduced experimentally to a low point of 80 degrees below zero, Fahrenheit, and will be able to obtain even lower temperatures if required. It is planned to enlarge the laboratory to include a hot room to simulate desert conditions and a wind tunnel for testing the permeability and wind resistance of fabrics designed for mountain and cold climate troops.

By means of controlled physiological and thermal measurements with human subjects, it will be possible in the new laboratory to make accurate tests of clothes or equipment, determine their suitability and otherwise evaluate their characteristics and effectiveness for the climate or conditions for which they are intended. With data supplied by these experiments, it will be possible to show where and when these items of clothing and equipment can be used.

Work of the Climatic Research Laboratory will be closely coordinated with the Climatic Research Unit in the Office of the Quartermaster General in Washington. This unit is drafting maps of world-wide climatic data and is studying the distribution and effects of cooling, evaporation and other allied climatological factors which would influence the effectiveness of America's fighting men in any part of the world. This eventually will include maps of insect prevalence, harmful animals, vegetation, etc.

The experiments will be conducted by physiologists, medical officers assigned by the Surgeon General and twenty volunteer enlisted men who will serve as controls in the tests and as laboratory assistants. The enlisted men are being selected from a large group who possess the educational and physical qualifications required and will be assigned to the laboratory on a semi-permanent basis. Their duties will include serving as test subjects, assisting in the installation and hand construction of equipment, reading instruments and recording research data, handling, storing and conditioning equipment and caring for the laboratory.

• CELANESE PATENTS

Two United States Letters Patents were granted to Celanese Corporation of America recently on applications of William Whitehead. Both patents relate to the production of laminated fabrics.

The first patent, No. 2,311,012, is directed to a process for the production of such fabrics. According to this process, a plurality of layers of fabric, at least one of which contains a plasticized derivative of cellulose, is united under heat and pressure, the union being effected in the presence of moisture applied to only one of the layers of fabric.

The second patent, No. 2,311,013, is directed to apparatus for manufacturing laminated fabrics according to the above process. This apparatus comprises a co-operating pressing head and buck for applying pressure on the fabric layers and, in addition, means for providing moistened fabric on the buck surface.

As a result of these patents, it is said to be possible to produce laminated and stiffened fabric webs and articles free from wrinkles and distortions and with superior adhesion between the several layers of fabric.

• MEETING, S.O.C.M.A.

The regular monthly meeting of the Synthetic Organic Chemical Manufacturers Association was held at the Chemists' Club, New York City on Wednesday, March 10th. The next meeting will be held on Friday, April 16th.

• APPOINTED LICENSEE

Guyan Mills, Inc., of Pawtucket, R. I., has been appointed a finisher licensee under the American Viscose Corporation's "Crown" Tested Plan, being fully qualified to dye and finish rayon fabrics in accordance with the "Crown" Tested minimum requirements, it was announced recently.

• INVISIBLE "RAINCOAT"

An invisible "raincoat" which can be formed on cloth, paper and many other materials by exposing them to chemical vapors from a new compound, thereby making them water-repellent, has been developed in General Electric's Research Laboratory at Schenectady, N. Y. by Dr. Winton I. Patnode who is studying many possible uses of this new method of water-proofing.

Called Dri-Film by the G. E. Electronics Department which will market the new compound, one of its most important uses so far is the treatment of ceramic insulators for radio equipment being made for the armed forces of the United States. It is claimed to be about nine times more effective than the wax used at present as a water repellent, and its results are permanent.

Dri-Film is a clear liquid composed of various chemicals which vaporize at a temperature below 100 C. Articles to be treated are exposed, in a closed cabinet, to the vapors for a few minutes. Then they are taken out and, if necessary, are exposed to ammonia vapor. This is to neutralize corrosive acids which may collect during treatment.

Dr. Patnode is not able to explain exactly what happens in the process, but the result is that an extremely thin film is formed on the surface. This "raincoat" is so thin that its structure cannot be determined by chemical analysis. It cannot be seen under a high-powered microscope. But, whatever its nature, it is said to prevent water from spreading to form a continuous film. If moisture does collect, it is in the form of small isolated drops.

• JOINS AMERICAN VISCOSE

Arthur J. Heinzmann has joined the American Viscose Corporation as an assistant in the "Crown" Tested Department, it was announced recently. Mr. Heinzmann was formerly connected with the Lawrence Print Works where he was superintendent of rayon dyeing and finishing, and prior to that was with Pacific Mills in charge of production of rayon dyeing and finishing. In his new position Mr. Heinzmann will work with Arthur R. Wachter in conducting the operation of the American Viscose Corporation's "Crown" Tested Plan, under which fabrics containing the company's "Crown" brand rayon yarn and staple fiber are tested for service qualities such as colorfastness, tensile strength and dimensional restorability.

• BOSTON OFFICE, HERCULES POWDER CO.

Hercules Powder Company has announced it will open sales offices in Boston on April

1, for its Cellulose Products, Naval Stores, Synthetics, Explosives and Paper Makers Chemical departments. The offices are on the seventh floor of the Statler Office Building.

Homer C. Simmons, New England manager of sales of the Cellulose Products Department, who is now at Stoneham, Mass., will head this department unit in Boston.

Howard C. Bates, in charge of Synthetics Department sales for that territory, will be located in the Boston office.

W. M. Williams, the newly-appointed resident manager of the Paper Makers Chemical Department's plant at Stoneham, will also head the Industrial Chemical Division sales group of the PMC Department in Boston.

G. J. Rising, former resident manager at Stoneham, will be transferred to the Hercules office in Pittsburgh, Pa., where he will represent the Industrial Chemical Division.

● REPELLENT TREATMENT FOR LAUNDRIES AND DRY CLEANERS

Treating clothes with a chemical making the garments shed water and resist stain soon may be a common operation for dry cleaners and laundries, the Fine Chemicals Division of E. I. du Pont de Nemours & Company announced recently.

Two improved water repellents developed by Du Pont—one for use with dry cleaning fluids and the other with wet washes—make readily available for civilian garments a protection which already is extensively employed in care of clothes for the armed forces.

Clothes are made water and stain resistant by dipping them in a solution after washing or dry cleaning.

It is stated that the chemical treatment of the fabric cannot be seen or felt, has no odor, and does not impair the appearance or draping qualities of the garment

A fabric made water repellent by these chemicals does not prevent normal evaporation from the human body as does a waterproofed fabric such as a rubberized raincoat. Treated garments usually clean more easily, saving time and preserving materials. Maintenance of the original properties of the fiber, conservation of clothes, and added health protection for those who must be outdoors in bad weather are among the advantages claimed for repellents.

"Aridex" L water repellent is the emulsion type which is diluted with water and applied in a laundry wheel after wet cleaning or washing. "Aridex" DCX is the solvent type which is diluted with the cleaning solvent and applied either in a standing bath or washer after dry cleaning. Both these and others in the "Aridex" line, which textile mills have used for several years to treat fabrics, are manufactured by the Du Pont Company and now are available in quantity.

● DIM OUT FABRIC

Celanese Corporation of America was recently granted United States Letters Patent No. 2,313,904, relating to the making of a textile fabric suitable for use for dim out purposes. One such type of material, "Celanese Dim Out Sheer," has been sold by the corporation during the past several months and is now in wide use in areas where dim out regulations are in force.

It is stated that when fabric made in accordance with the invention is used as dim out material in display windows, the glare of the illumination is effectively eliminated.

The invention also includes the use of the fabric for dimming out light not only in the case of display windows, but for all other dim out purposes.

In order that the desired anti-glare effect be attained, the fabric should be of a very dark color. Preferably, it should be dyed black but may be of navy blue or dark brown.

● MARCH ISSUE, TEXTILE RESEARCH

Development of the first successful continuous process for dyeing woolen piece goods with the fast indigo required by the United States Navy, is described in the March issue of *Textile Research,* official publication of Textile Research Institute, Inc., and of the Textile Foundation. In this article is related the story of the research and engineering that made this development a success—a development which represents one of the major contributions by the textile industry to production for war requirements. It is another outstanding accomplishment of research over a long period, followed by a period of close co-operation between a textile mill and the research laboratory of a dye manufacturer which made it possible to develop in a few months time, under pressure of necessity, what would ordinarily have required years.

Drying of textiles at higher temperatures than had previously been thought possible is expected as a result of researches on the proper relationship of temperature, relative humidity of the heated air, and time of exposure. The results of these studies, which were made in connection with the drying research project of the Textile Research Institute, Inc., make it possible to form some practical estimates of the limiting conditions of temperature, moisture, and time of exposure, in the drying of textiles.

Among other features of the March issue of *Textile Research* is an article describing the results of tests made on blankets constructed of part-wool and part-cotton. From the data secured in these tests it was concluded that part-wool blankets would be a satisfactory substitute for the all-wool Army blankets for use in barracks and effect a substantial saving in wool.

● NYLON WINDOW SCREEN

An entirely new window screen, chemically made, and containing no metal, will be available to American householders after the war.

It is made of nylon, now used exclusively for vital military purposes. It is claimed that, having all of the good characteristics of metal screening, and many qualities besides, it can be produced in any color, it will not stain the sills, it will not corrode, it requires no painting, and tests indicate it has extraordinary durability. Pencils or other sharp-pointed objects can be shoved through it without damage; the strength and elasticity of the strands is said to be so great that they come back into place merely by rubbing them with one's fingers.

In many cases the new screens will not even have to be put up in the spring and taken down in the fall. They will just be rolled up and down on tracks like a window shade.

During the coming summer wide-scale tests of the new screens will be made looking toward military applications and as a guide to further development of the product.

Active work on the screening got under way late in 1940 and during the following months the development continued with considerable technical success, until our entrance into the war called nylon into service for parachute cloth.

Meanwhile a few screens had been erected at various points for preliminary tests. These indicated that nylon was a promising material for this use. It is stated that they stood up well even along the seashore, where salt spray rusts or corrodes metal screens very rapidly. Nylon screen doors installed in Cuba in January, 1941, were said to be still "as good as new" a year and a half later, when they were taken down for "accelerated" weathering tests in the Nylon Research Laboratory back in Wilmington, Del.

First step in the manufacture of the synthetic screen is the spinning of the "wire." The raw material is very similar to that used to make nylon stocking yarn. However, before the nylon chips or flakes are put into the spinning hopper they are pigmented, and when the material is melted at the lower end of the hopper it flows into the spinneret as a uniformly colored viscous material, about the consistency of molasses. The "wire" is thus colored through and through and the color is said to last as long as the "wire" does.

Recent Advances in

FUR DYEING*

Improved Results with Oxidation Colors: Use of Peroxidase

DR. H. WEISSKOPF

OXIDATION dyestuffs, i.e., those coloring matters which are characterized by the fact that they are not added in substance to the dyebath but are formed and deposited on the fiber by oxidation of an intermediate during the dyeing process, are not of great importance in textile dyeing, with the notable exception of aniline black, but they are the very foundation of the modern fur dyeing industry. The present article reviews this method of coloring furs and mentions some recent efforts to obtain improved results from oxidation dyes.

The bases which are oxidized to dyestuffs are monocyclic aromatic amines and phenols. In recent years, however, Kozlov and Zhuk[1] have investigated the suitability of corresponding naphthalene derivatives, and some references to this subject are to be found in patent literature. The oxidation of the bases is carried out in aqueous solution by hydrogen peroxide. Dyeing is performed either by the dipping method, which consists in immersing the material to be dyed into the solution until the oxidation is complete, or by applying the solution to furs and human hair by means of a brush or spraying apparatus. The brush (or spraying) method is known in the fur trade as "blending."

The chemistry of the formation of the coloring principle is generally the same as in the formation of aniline black. But the latter process requires one final product only, namely deep black, whereas in fur dyeing a large range of shades must be obtained, varying from brightest beige to deepest black, and including grey, bluish and brown shades. Each of the desired shades requires a corresponding oxidation product, which means a special oxidation reaction. These variations are obtained by application of different dyeing bases.

SUITABLE BASES

Of the variety of bases suitable for this purpose the most important ones are:—

(a) *Amines.* p-aminodimethylaniline, p-phenylenediamine, aminodiphenylamine, m- and p-toluylenediamine.

(b) *Aminophenols.* o- and p-aminophenol, 2: 4-diaminophenetol.

(c) *Phenols.* catechol, resorcinol, pyrogallic acid. Nitro- and halogen-substituted derivatives of the compounds mentioned under (*a*) and (*b*) are also used.

The first stage of oxidation for all amine bases is the formation of quinoneimines, which in the second stage form indamines and indophenols. The reaction does not, of course, stop at this point, indamines and indophenols being unstable and sensitive to acids. Forming a ring system they change into phenazines and oxazines.

The process is, as already mentioned, similar to the formation of aniline black, but special problems arise as a result of the variety of bases, their chemical constitution and the dyeing methods for furs and human hair, which differ greatly from textile dyeing. Furs, for instance, may not be dyed in boiling or even hot baths, because temperatures higher than 35° C. cause damage to the leather.

REACTIONS OF P-PHENYLENEDIAMINE

The first dyeing base, which was patented in 1888 by Erdmann, was p-phenylenediamine. As it is still one of the most important bases, its oxidation reaction can be profitably considered more closely. The first oxidation product of p-phenylenediamine is quinonediimine, which is very unstable and polymerizes itself to the so-called Bandrowski's base. The result of this process is a tri-nucleus compound (with two benzeneoid nuclei and one quinonoid nucleus), which derives from three quinoneimines.

By keeping quinonediimine in water solution Willstaetter obtained a compound similar to Bandrowski's base. This base crystallizes in brown needles and is soluble with difficulty in water.

p-phenylenediamine dyes animal hair in shades ranging from reddish-grey to blue-violet, which means that Bandrowski's base, if it appears at all, as a result of oxidation, does so only along with other oxidation products, forming a mixed color. Animal hair dyed directly with Bandrowski's base gives a dirty yellow color. Whereas Bandrowski's base is scarcely soluble and thus the amount deposited on the fiber in the foregoing experiment is only a small one, nevertheless the experiment confirms the above-mentioned assumption that compounds other than Bandrowski's base are formed when oxidizing p-phenylenediamine under dye house conditions. These compounds

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* Reprinted from *The Dyer and Textile Printer.*

are due probably to ring-closure, their molecules being larger than those of Bandrowski's base*

Under dyehouse conditions p-phenylenediamine is never used alone, but only in admixture with other bases, and the same may be said of all other fur dyestuffs. In this connection, the task of the phenols listed under (c) must be mentioned. They are not satisfactory for "self" shades like the amines, but as "developers" combined with amines they are of importance in the formation of the intermediate stage of indophenols.

For a long time the dyeing bases under consideration in this article have been successfully applied in biological research in order to prove the existence of oxidative catalysis, i.e., oxidation enzymes. The proof is given by appearance of intensely colored oxidation products. For instance, p-phenylenediamine is used for detection of cellular oxidases and, in combination with hydrogen peroxide, of peroxidase, especially for milk. In the terminology of enzyme chemistry these bases are "chromogenic compounds," capable of being dehydrogenated in presence of H_2O_2. Such a system is activated strongly by peroxidase.

INTENSIFIED OXIDATION

It has therefore been the aim of one of my investigations[3] to find out whether by means of intensified oxidation caused by peroxidase, an improvement of technical dyeing can be obtained in respect to fastness of colors, and saving of dyestuff and oxidation agent. The dyeings of o-and p-aminophenol, benzidine, catechol and pyrogallic acid in presence of peroxidase show a deepening of color compared with normal dyeings. Only dyeings of p-phenylenediamine (in presence of peroxidase) show a brighter color and are similar to the above-mentioned dyeing obtained by means of Bandrowski's base. It may therefore be assumed that in the case of intensely activated oxidation, p-phenylenediamine forms Bandrowski's base, i.e., the quinonediimine previously formed apparently polymerizes quantitatively. In cases of mixtures of dyeing bases, as are used in practice, I was able to obtain intensification of coloring up to 20 per cent, through the influence of peroxidase. Further experiments on a large scale must be made, however, if it is to be proved that the application of these findings can be of advantage to the dyeing industry.

In practical dyeing the mordants and the pH of the baths are of great importance. Fastness to light and storage are increased by means of dyeing on material mordanted by Fe, Cu, and Cr salts. Generally, mordants cause an intensification of the color. In the case of Fe and Cu salts as mordants this intensification is probably caused not only by formation of a colored lake, but also because the heavy metal salts have an activating effect on the oxidation. In this connection it may be pointed out that Wertheimer[3] has proved the existence of "heavy metal catalysis" by Cu salts in the formation of indophenol blue from p-phenylenediamine + *a*-naphtol.

The shade of dyeing very largely depends on the pH of the baths[4].

The question arises whether the pH directly influences the mechanism of oxidation or whether the oxidation product may be of different color according to whether it is a free basic compound or a mono- or polyacidic salt. The experiences with oxazine and phenazine compounds, of which differently colored salts are known, are in favor of the last mentioned assumption. The molecular size of the oxidation products being remarkable, it may be assumed that the degree of dispersion is effected by pH. As regards the sols of emeraldine, Ptshelin[5] has proved this connection, and since changes of degree of dispersion are generally accompanied by changes of color, this possibility must be taken into consideration when explaining dependance of coloring on pH.

MISCELLANEOUS

A group of dyestuffs has been proposed which do not contain p-phenylenediamine and can be dyed in admixture, as the different bases do not react with one another. It is only on mordants that they give a color of sufficient intensity; their solutions are colloidal. These qualities speak in favor of the assumption that the compounds mentioned have a greater molecular size than simple bases. They very likely represent a higher degree of oxidation of the simple bases. Patents were granted for the use of diazotization in the following manner: the bases hitherto used are dyed as usual and diazotized after completed oxidation. In this way an improvement of fastness is said to be attained.

These various proposals have not yet had much acknowledgment in the fur dyeing industry, where dyeing is generally carried out according to the "classical" methods discussed in this article.

In hair dyeing one is faced by a special problem: quinonediimine has toxic qualities and may impair the health of some persons (dermatitis). Efforts have therefore been made to avoid p-phenylenediamine in mixtures destined for hair dyeing. It is substituted by p-amino-piphenylamine, for example, which may be regarded as leuco-compound of an indamine. From this leuco-compound the indamine is derived (being the first oxidation product of the leuco-compound) without the intermediate appearance of quinonediimine. By using 4-aminophenylglycine as dyeing base the appearance of quinonediimine may again be avoided.

REFERENCES

[1] *J. Applied Ch.* (U.S.S.R.), 12, 1501 (1939).

[2] No. 2, vol. 1, *Journ. of Central Scientif. Research Labor. of Fur Ind. Trust, "Sojusmekhprom,"* Moscow, 1933.

[3] *Fermentforschung*, VIII., 497 (1925).

[4] *Chem. Z*, vol. 1930, p. 600.

[5] *Koll. Z*, 70, 306 (1935).

* It may be supposed that quinonediimine condenses with p-phenylenediamine not yet oxidized to an indamine, which undergoes further condensation.

MICROSCOPICAL IDENTIFICATION

of Lanital, Aralac and Soybean Fibers

G. L. ROYER

Research Department, Calco Chemical Division, American Cyanamid Company

DURING the preparation of a motion picture entitled "Wool Dyeing Phenomena," a brief description of which has been published[1], it was found that the reaction of concentrated sulfuric acid with the casein fiber known as Lanital revealed a phenomenon which might be useful as an identification test. Figure 1 is an enlargement of a single frame from this movie illustrating the phenomenon. Note the diamond shapes which form. Further investigations[2, 3] have shown that this test is also useful for the identification of the synthetic protein type fibers, Aralac and Soybean. Photomicrographs of these can be seen in Figures 2 and 3. This phenomenon has not been found to occur in any of the following fibers: cotton, wool, cellulose acetate, Bemberg, Viscose, cisalfa,

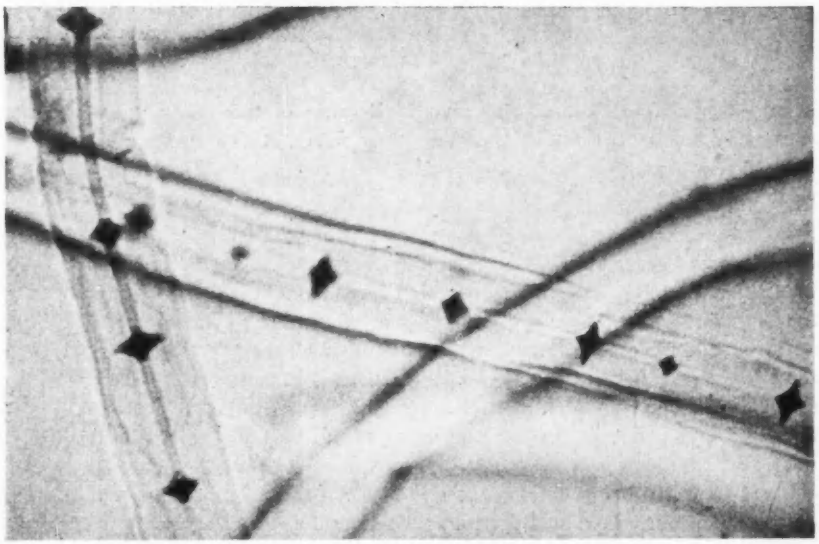


Figure 1

LANITAL

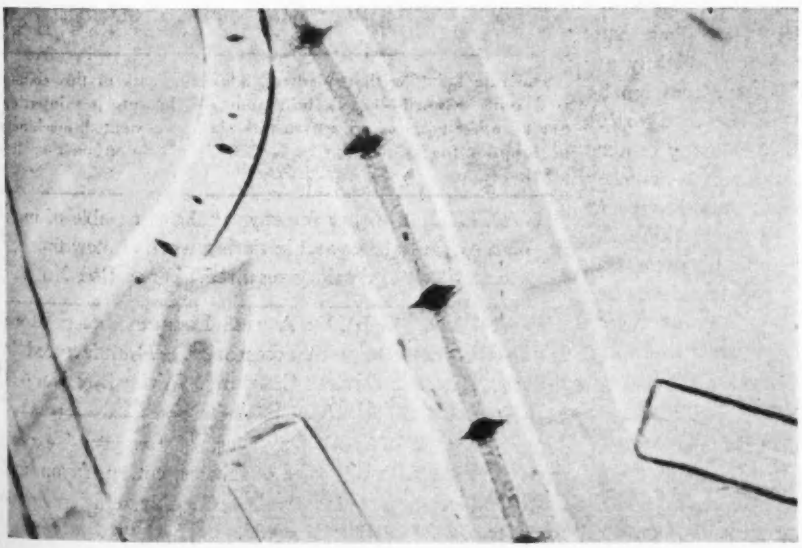
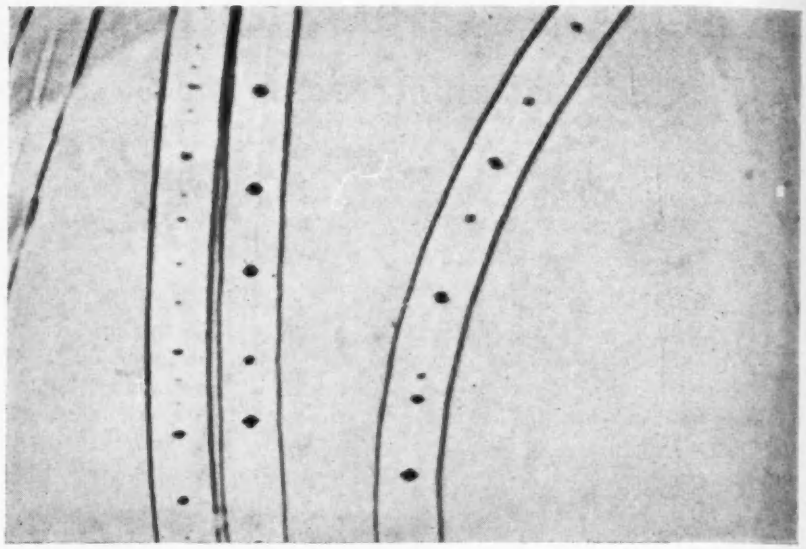


Figure 2

ARALAC

Figure 3

SOYBEAN



nylon, silk, slovena, Tubize or Vinyon. Because of the increased use of the synthetic protein type fibers, it is thought that a complete description of the test would be of value.

In this test the dry fibers are teased apart and placed under a cover glass on a slide under the microscope. A 20X objective and a 10X or 20X occular give a magnification suitable for observation. The concentrated sulfuric acid is introduced under the cover glass by placing a drop on the slide at the edge of the cover glass. The acid runs under the slide partly aided by capillary flow and wets the fibers. The acid penetrates into the individual fibers and the effect of the acid can be followed by means of the changes in index of refraction at the diffusion front. If dyed fibers are used the acid destroys or dissolves the dye and this aids in making the diffusion front more prominent. Just before complete diffusion is reached, a narrow strip remains unpenetrated in the center of the fiber. At this time the fiber suddenly seems to contract slightly at certain places along the sides and at these places small well formed diamonds appear within the fiber. Shortly thereafter, that is as the acid penetrates to the very center of the fiber, the diamonds slowly close again to cracks perpendicular to the edges of the fiber and then disappear entirely. The diamonds appear and disappear quickly and care must be taken to watch constantly or the phenomenon may be overlooked. They seem to occur at somewhat regular intervals along the fibers. It is believed that these diamonds form from the stress in the fiber when the fiber is partially softened by the acid. Complete softening of the fiber releases the stress and the figures disappear.

Tests with lower concentrations of acid on Aralac, Lanital and Soybean fibers show that the diamonds form at different times after adding the acid depending both upon the type of fiber and the acid concentration. It has been possible to differentiate between the different types of synthetic protein fibers in the laboratory on this basis. However, it must be kept in mind that changes in the manufacture of the same fiber may effect this phenomenon.

In addition to the use of this test as an identification, it might be useful for a study of the structure and stability of the synthetic protein fibers. Changes in the fiber structure to effect better wet strength and stability to alkali might be followed by a more complete examination of this phenomenon.

[1]Kienle, R. H. and Royer, G. L. American Dyestuff Reporter *28*, 638 (1939).
[2]Millson, H. E. Calco Technical Bulletin 667 issued October 15, 1942.
[3]Haller, H. C. American Dyestuff Reporter *31*, 681 (December 1942).)

•CLASSIFIED• ADVERTISEMENTS

The rate for "Position Wanted" advertisements in this column is 2 cents a word—with a minimum of 50 cents per insertion. For all other types of advertisements—i.e., help wanted, machinery or supplies for sale—the rate is $6.25 per column inch or less per insertion.

WANTED: Finisher for rayon fabrics capable of making up his own finishes and initiating work on new finishes. State experience and salary required. Write Box No. 429.

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BULLETIN NO. 635 — Supplement No. 1: "Dyeing of Herringbone Twill O. D. No 7, O. D. No. 8, Khaki No. 1"

BULLETIN NO. 674: "Dyeing of Sulphon Cyanine Blues"

BULLETIN NO. 676: "Nylon"

BULLETIN NO. 679: "Dyeing of War Aid Olive Drab Worsted Knitting Yarn"

BULLETIN NO. 681: "Piece Dyeing With Sulfur and Vat Dyes of Work Clothing"

BULLETIN NO. 682: "Dyeing and Finishing of Mosquito Netting"

BULLETIN NO. 683: "The Coloring of Camouflage Shrimp Nets"

BULLETIN NO. 685: "Olive Drab. Package Dyeing of Mercerized Yarn for Knitted Underwear"

BULLETIN NO. 687: "The Electron Microscope and Cellulose"

BULLETIN NO. 689: "Dyeing of Toweling — Olive Drab No. 3—Turkish and Terry"

BULLETIN NO. 691: "Cloths, Wool, Covert—14 oz."

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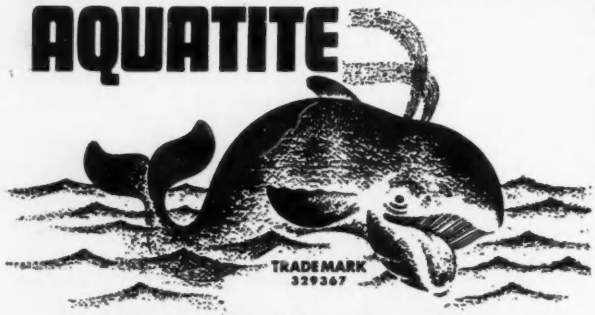

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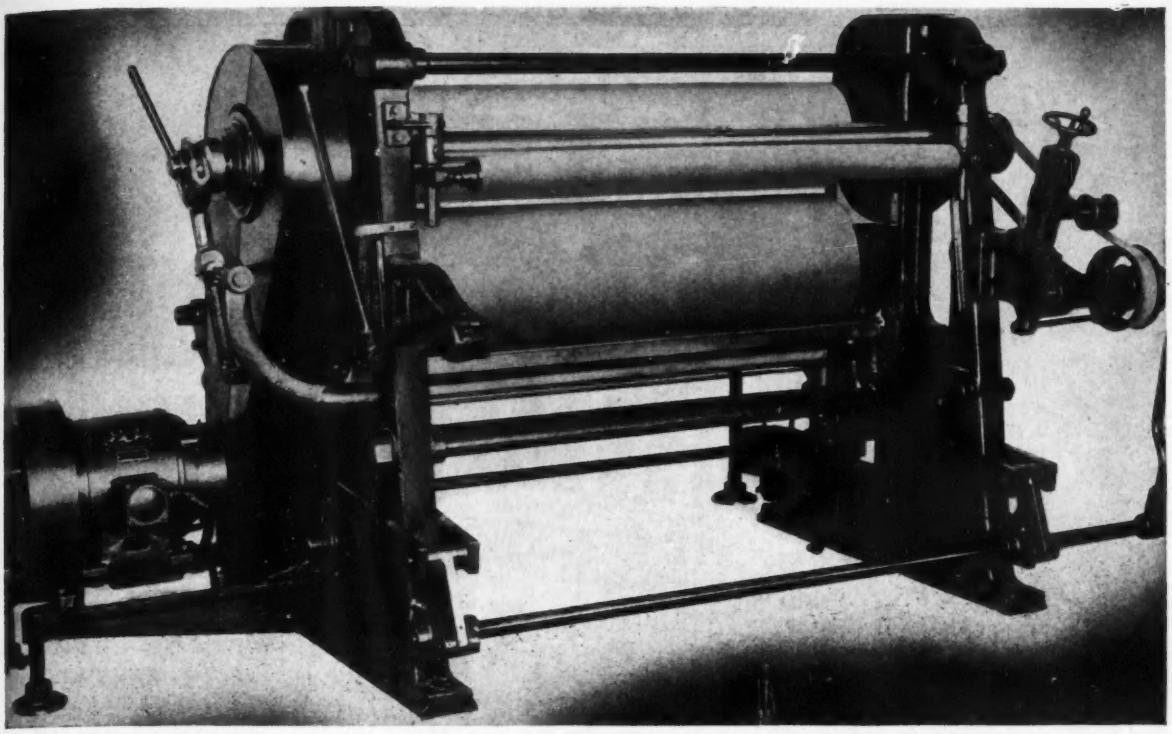

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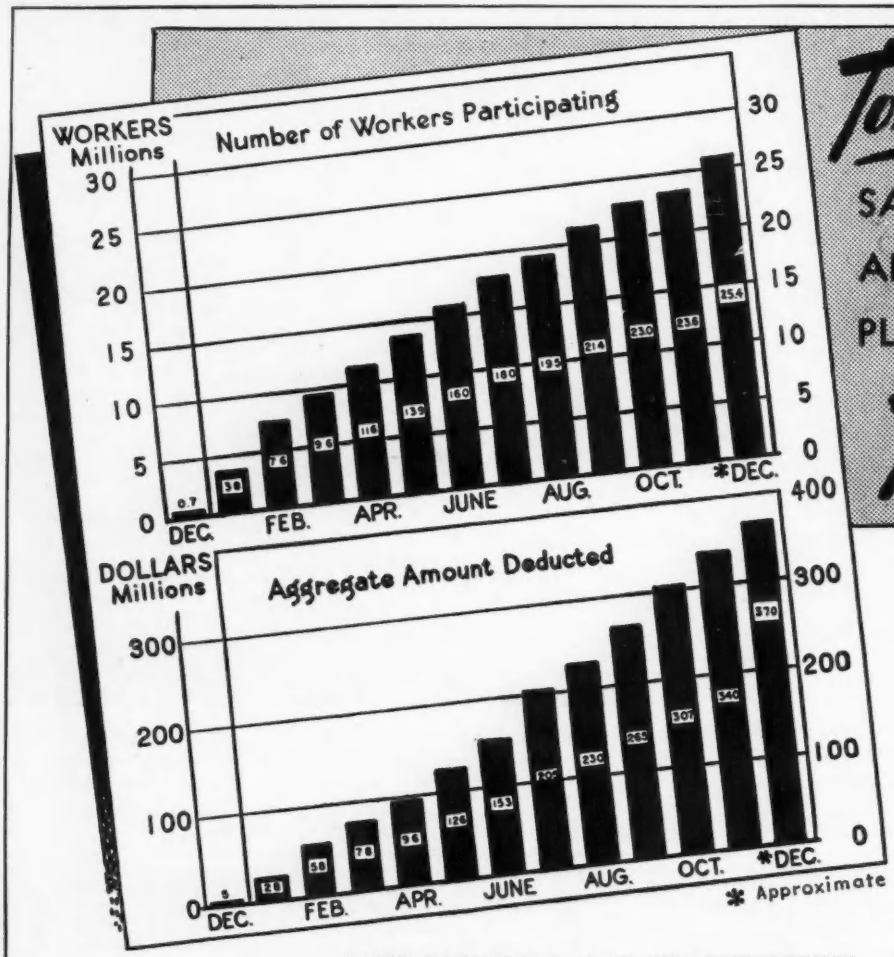

Tomorrow's SALES CURVES ARE BEING PLOTTED . . . Today

THESE CHARTS SHOW ESTIMATED PARTICIPATION IN PAYROLL SAVINGS PLANS FOR WAR SAVINGS BONDS *(Members of Armed Forces Included Starting August 1942)*

STUDY THEM WITH AN EYE TO THE FUTURE!

There is more to these charts than meets the eye. Not seen, but clearly projected into the future, is the sales curve of tomorrow. Here is the thrilling story of over 25,000,000 American workers who are today voluntarily saving close to FOUR AND A HALF BILLION DOLLARS per year in War Bonds through the Payroll Savings Plan.

Think what this money will buy in the way of guns and tanks and planes for Victory today—*and mountains of brand new consumer goods tomorrow.* Remember, too, that War Bond money grows in value every year it is saved, until at maturity it returns $4 for every $3 invested!

Here indeed is a solid foundation for the peace-time business that will follow victory. At the same time, it is a real tribute to the voluntary American way of meeting emergencies that has seen us through every crisis in our history.

But there is still more to be done. As our armed forces continue to press the attack in all quarters of the globe, as war costs mount, so must the record of our savings keep pace.

Clearly, on charts like these, tomorrow's Victory —*and tomorrow's sales curves*—are being plotted today by 50,000,000 Americans who now hold WAR BONDS.

EVERYBODY EVERY PAYDAY AT LEAST 10%

Save with

War Savings Bonds

This space is a contribution to America's all-out war effort by

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All in controllable cycles inside the

NEW ATLAS TWIN ARC WEATHER-OMETER

TEST RESULTS TWICE AS FAST

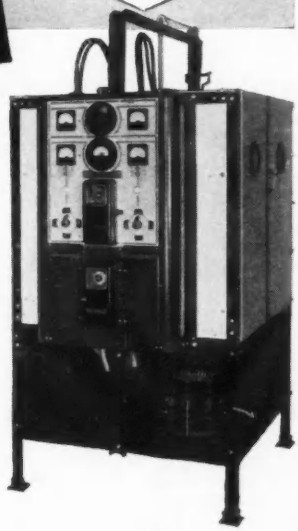
THE new Atlas Twin-Arc Weather-Ometer has been developed to save time where speed in testing is essential.

Where Federal Specifications call for a definite number of testing hours in the Atlas single arc Weather-Ometer, this time can be cut in half in the Atlas Twin-Arc.

Exclusive Atlas Twin-Arc Weather-Ometer features:

1. Temperature control.
2. Unlimited range and control of Light and Water Spray Periods.
3. Adjustable and automatic timing.
4. Insulated test chamber.
5. Operates continuously 24 hours without manual attention.
6. Carbon cost 28c per day.
7. Full automatic — Safe to operate unattended overnight.
8. Control panel contains Volt and Ammeters—Time Meter—Light and Water Cycle switch—Automatic time cut off switch—Voltage adjusting switch—Direct reading thermal regulator—Reactance Coil (cuts power cost in half).

The Single Arc Model is a popular machine where high speed is not required.



Originators and sole manufacturers of accelerated testing devices for a quarter of a century. Fade-Ometer, Weather-Ometer, Launder-Ometer are used all over the world as accepted standard testing machines.

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361 W. Superior St., Chicago, Illinois

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Why so many grades? Because the properties of the various silicates are different and produce different effects, they fill many needs in the textile industry, e.g.

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Lengthens life of bleach bath

KIER BOILING . . . . . "TN" Brand.
Improves removal of wax and oil

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Prevents staining of cloth

RAYON SCOURING . . . . Metso Granular.
Cleaner fabrics, more level dyeing

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Produces a water-repellent finish on uniform cloth, overcoatings, and other military fabrics . . . used in a single bath treatment with Fungicide G to impart water-repellency and mildew resistance . . . used in a single bath treatment with Arko Fire Retardant to produce water-repellency and fire-resistance.

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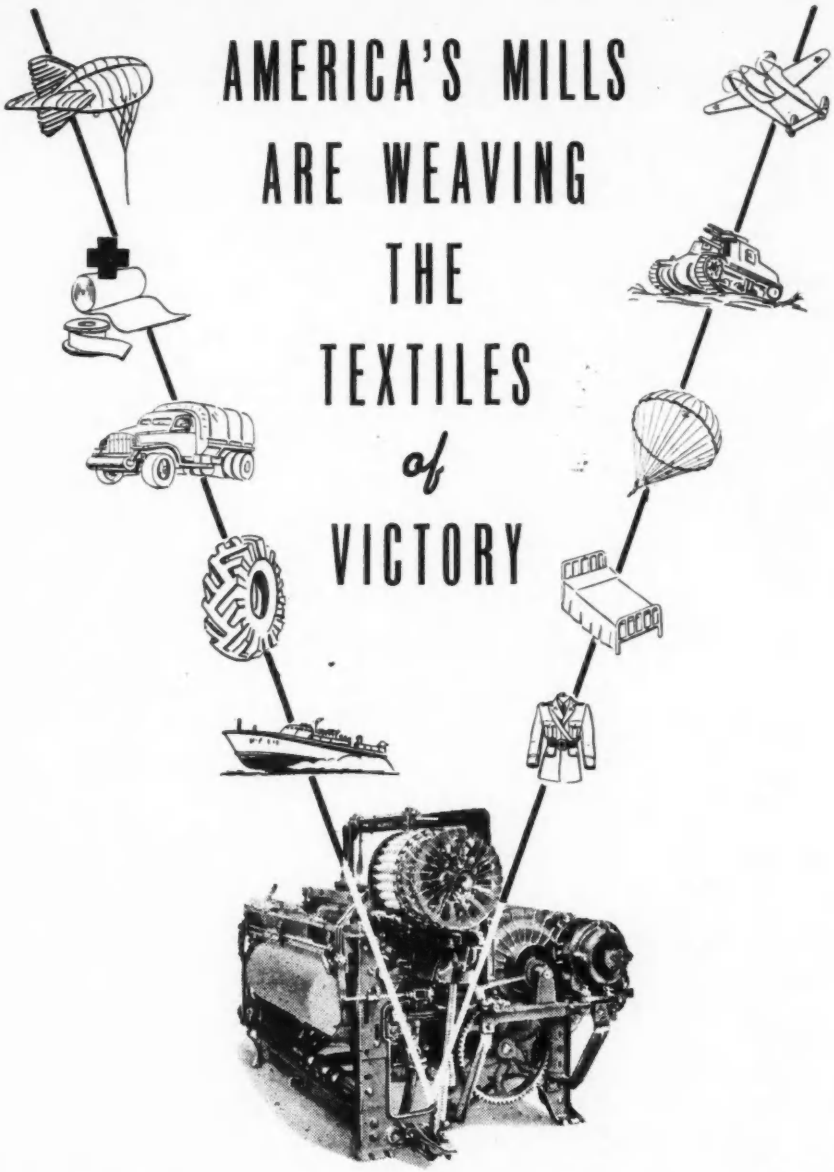
• INDEX • TO ADVERTISERS

Alco Oil & Chemical Corp.	XVI
Althouse Chemical Co.	
Amalgamated Chemical Corp.	
American Aniline Products, Inc.	III
American Cyanamid & Chemical Corp.	Back Cover
American Dyewood Co.	
American Viscose Corp.	XI
Ansul Chemical Co.	
Aqua-Sec Corp.	
Aridye Coro.	IX
Arkansas Company, Inc.	XX
Arnold-Hoffman & Co., Inc.	
Atlas Electric Devices Co.	XIX
Becco Sales Corp.	
Bick & Co., Inc.	
Blickman, Inc., S.	
Burkart-Schier Chemical Co.	XXI
Butterworth & Sons Co., H. W.	
Calco Chemical Division, American Cyanamid Co.	XV
Campbell & Co., Inc., John	
Carbide & Carbon Chemicals Corp.	
Carbic Color & Chemical Co., Inc.	XII
Ciba Company, Inc.	
Colgate-Palmolive-Peet Co.	
Commonwealth Color & Chemical Co.	XVII
Du Pont de Nemours & Co., Inc., E. I.	
Dyestuffs Division	Front Cover
Electrochemicals Dept.	
Graselli Chemicals Dept.	
Emery Industries, Inc.	
Exact Weight Scale Co.	
Fancourt & Co., W. F.	
Geigy Company, Inc.	
General Dyestuff Corp.	VII
Hart Products Corp.	
Hercules Powder Co.	
Naval Stores Department	
Synthetics Department	
Hooker Electrochemical Co.	
Industrial Rayon Corp.	VIII
Kali Manufacturing Co.	
Kelco Co.	X
Laurel Soap Mfg. Co.	
Mathieson Alkali Works, Inc.	
National Aniline Division, Allied Chemical & Dye Corp.	XIV
National Carbon Co., Inc., Carbon Sales Division	
Nyanza Color & Chemical Co.	
Onyx Oil & Chemical Co.	VI
Perkins & Son, B. F.	
Philadelphia Textile Finishers, Inc.	
Philadelphia Quartz Co.	XIX
Prince George Hotel	
Procter & Gamble	
Refined Products Corp.	IV, V
Rohm & Haas Co.	XXII
Royce Chemical Co.	
Sandoz Chemical Works	
Scholler Bros., Inc.	Third Cover
Solvay Sales Corp.	
Stein, Hall & Co., Inc.	
Union Carbide & Carbon Corp.	
Van Vlaanderen Machine Co.	XVII
Virginia Smelting Co.	
Westvaco Chlorine Products Corp.	
Wallerstein Co., Inc.	XVI
Warwick Chemical Co.	Second Cover
Wolf & Co., Jacques	
Young Aniline Works	
Zinsser & Co.	

RS
XVI
III
k Cover
XI
IX
XX
XIX
XXI
XV
XII
XVII
Cover
VII
VIII
X
XIV
VI
XIX
IV, V
XXII
Cover
XVII
XVI
Cover
PORTER

AMERICA'S MILLS ARE WEAVING THE TEXTILES *of* VICTORY



MORE than 300 pounds for every man in the Services! That's the staggering amount of textiles being supplied to our armed forces right now.

Clothing, of course, makes up part of that poundage—but only part. For there is scarcely a piece of equipment made that does not use textiles somewhere in its construction. From lifeboats to army jeeps, from tanks to planes, from parachutes to camouflage—textiles are on active duty.

Burkart-Schier is helping to make possible the production record being set by the textile industry—the mile after mile of needed fabrics which are speeding off America's looms. For Burk-Schier chemists and textile chemicals are working right along with the textile industry, helping to process all sorts of military fabrics, giving fabrics necessary characteristics, making fabrics fit to fight!

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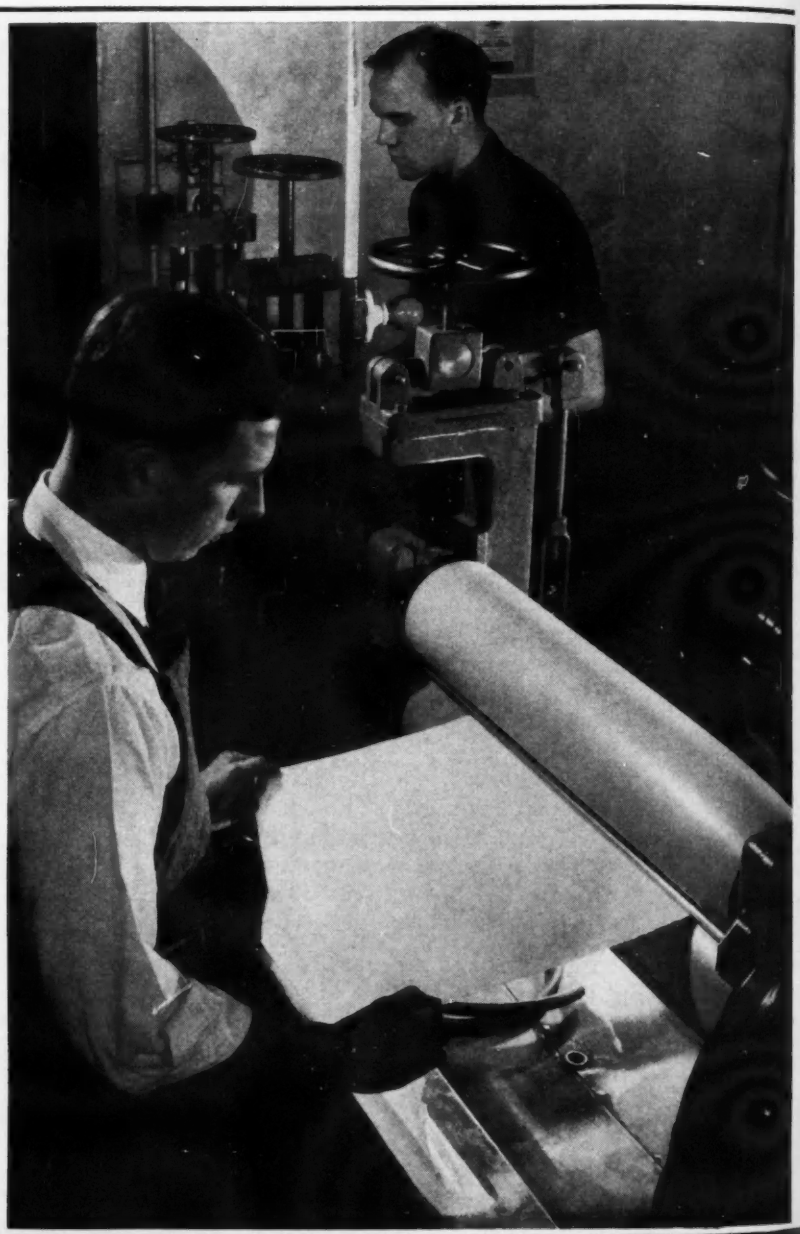

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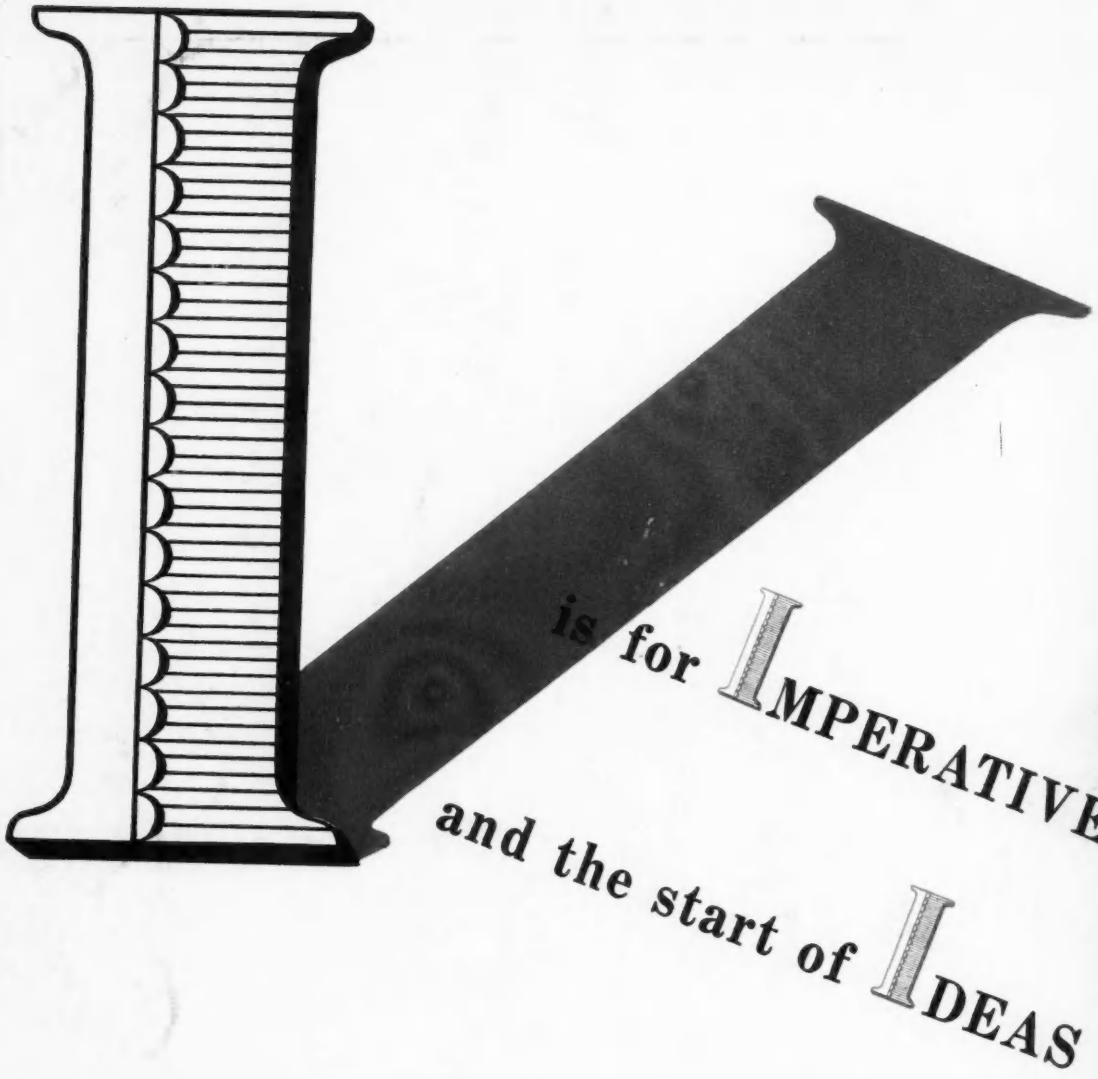

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